

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date
13 May 2004 (13.05.2004)

PCT

(10) International Publication Number
WO 2004/039916 A1

(51) International Patent Classification⁷: **C09K 21/06**, C01B 33/44, C08K 3/34

(21) International Application Number: PCT/AU2003/001443

(22) International Filing Date: 31 October 2003 (31.10.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data: 2002952373 31 October 2002 (31.10.2002) AU

(71) Applicants (for all designated States except US): COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION [AU/AU]; Limestone Avenue, Campbell, Australian Capital Territory 2612 (AU). THE BOEING COMPANY [US/US]; 7755 East Marginal Way South, Seattle, WA 98108 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): ANGLIN, Matthew, Allen [US/US]; 6403 Linden Avenue North, Seattle, WA 98103 (US). BATEMAN, Stuart, Arthur [AU/AU]; 3 Wortley Avenue, Mount Waverley, Victoria 3149 (AU). MACLEAN, Barbara, Lord [US/US]; 9824-NE 13th Street, Bellevue, WA 98004 (US). PARTLETT, Matthew, Jon [AU/AU]; 1/69-71 Noble Street, Allawah, New South Wales 2218 (AU). PETERSON, James, Macon [US/US]; 420 SW 183rd Street, Seattle, WA 98166-39342 (US). WU, Dong, Yang [AU/AU]; 6 Arnott Court, Wheelers Hill, Victoria 3150 (AU).

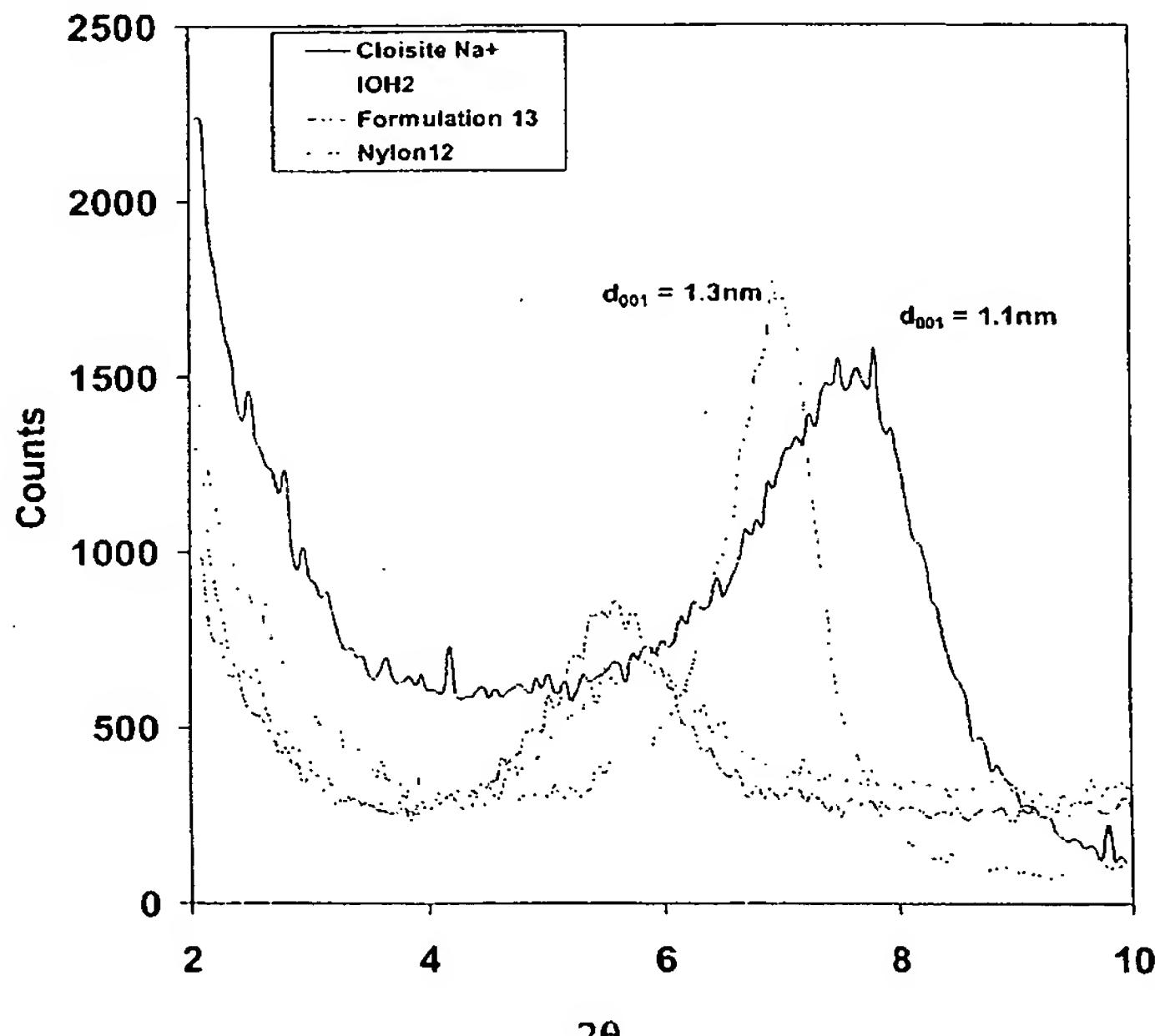
(74) Agent: GRIFFITH HACK; 509 St Kilda Road, Melbourne, Victoria 3004 (AU).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (regional): ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),

[Continued on next page]

(54) Title: FIRE RESISTANT MATERIAL



(57) **Abstract:** The present invention relates to inorganic-organic hybrids (IOHs), methods for their preparation and their use as fire resistant materials or components of fire resistant materials. More specifically, the invention relates to polyamide fire resistant formulations containing IOHs which have application in the production of fire resistant articles or parts thereof for use in the transportation, building, construction and electrical or optical industries.

WO 2004/039916 A1

Rec'd PCT 02 MAY 2005

WO 2004/039916 A1



European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

Published:

- *with international search report*

FIRE RESISTANT MATERIAL

The present invention relates to inorganic-organic hybrids (IOHs), methods for their preparation and their use as fire resistant materials or components of fire resistant materials. More specifically, the invention relates to polyamide fire resistant formulations containing IOHs which have application in the production of fire resistant articles or parts thereof for use in the transportation, building, construction and electrical or optical industries.

BACKGROUND OF THE INVENTION

Materials based on organic polymeric systems (plastics) are widely used in the transportation, building and construction industries. A drawback of many types of organic polymers is flammability which limits their suitability in applications requiring flammability resistance and where regulatory authorities govern flammability standards.

In commercially produced polymeric systems, flame-retarding species may be added during processing or forming of the materials to reduce the end products flammability. Conventional flame-retardants may be divided into different categories including:

Halogen based: which consist of either brominated or chlorinated chemicals such as brominated polystyrene or phenylene oxide (Dead Sea Bromine or Great Lakes CC) or bis(hexachlorocyclopentadieno) cyclooctane (Occidental CC).

Phosphorus based: which consist of a range of different chemistries from elemental phosphorus (Clariant), phosphonates (A&W antiblaze 1045), phosphonate esters (Akzo Nobel), phosphites, phosphates and polyphosphates including melamine phosphite and phosphate, ammonium and melamine polyphosphate (DSM Melapur).

- 2 -

Nitrogen based: such as melamine and its salts (US 4,511,684 Schmidt & Hoppe).

5 *Intumescent agents:* incorporating (i) an acid source (carbonization catalyst) such as ammonium polyphosphate; (ii) a carbonization reagent e.g. polyhydric alcohols such as pentaerythritol; and (iii) a blowing reagent like melamine. Expandable graphite is also known to undergo thermal expansion on addition of heat.

10 *Inorganic additives:* such as magnesium hydroxide and aluminum hydroxide (Martinswerk), zinc borate (Fire Brake ZB, US Borax) and antimony trioxide,

15 Although the addition of fire retardants to polymeric systems may improve their fire performance other important properties are often adversely effected for example:

- Mechanical performance
- Surface finish
- Durability
- Rheology
- Stability
- Smoke generation
- Toxicity
- Cost
- Recyclability

20 Furthermore, there has been considerable recent impetus to reduce the use of some flame-retardant classes due to toxicological or environmental concerns. Such legislation has placed pressure on the use of halogenated compounds and certain metal oxide synergists. Phosphorus-based flame-retardants such as phosphonates and elemental (red) phosphorus are also undesirable due to their regulation under chemical weapon acts and considerable manufacturing danger.

25 As far back as 1965, Jonas (GB 1,114,174) teaches that the incorporation of organically modified clay into plastics reduces melt dripping during combustion.

- 3 -

More recently it has been shown that under certain synthetic or processing conditions, organically modified clay may be nano-dispersed into polymeric materials to improve mechanical and fire performance.

5 Okada et al, (US 4,739,007 (1988) Toyota) teaches that nylon 6 materials with improved mechanical and heat distortion temperature can be prepared by adding suitably modified clay during the synthesis of nylon 6. In this case the growing nylon chains force apart the clay 10 platelets to form intercalated or exfoliated nanomaterial structures (so called in 'situ polymerisation' method).

A more commercially desirable method of nano-dispersing modified clay is described by Maxfield, et al, (WO 93/04118 WO 93/04117 (1993) Allied Signal). Maxfield 15 teaches that clay-plastic nanomaterials with improved mechanical and heat distortion performance may be prepared by subjecting functionalised clay and molten plastics such as nylon6, nylon66 and PBT to shear forces.

Others have investigated the fire performance of 20 plastics incorporating clay nano particles. Gilman has studied the fire performance of nylon-nanomaterials prepared through the 'in situ' polymerisation pathway using cone calorimetry (Proc. 43. Int. SAMPE Sympos., (1998), p1053-1066, Fire and Materials, 24, (2000), p201- 25 208, Applied Clay Science, 15, (1999), p31-49). Improved heat release rates were achieved with the addition of commercially modified clay, without increasing toxic gas or smoke generation. Gilman teaches that the improved fire performance results from the nanoparticles both 30 mechanically stabilizing the char and enhancing its barrier properties. Although Gilman's cone calorimetry tests suggest improved performance in terms of a reduction in heat release rate, no mention was made of other aspects of the materials fire performance in common tests 35 described by bodies such as ASTM and FAA which are used to assess, regulate and qualify the fire worthiness of materials.

- 4 -

Other groups have reported that traditional flame-retardants and nano-dispersed clays can act synergistically to improve fire performance.

Klatt (WO 98/36022, (1998) BASF) teaches that 5 nylon materials incorporating organically modified clay and red phosphorus synergistically improve fire performance to produce a VO rating in UL94 type vertical burn tests. However, such compositions are undesirable due to the danger associated with handling of elemental 10 phosphorus.

Morton (WO 99/43747, (1999) General Electric Company) teaches that in certain polyester blends, phosphorus based flame retardants especially resorcinol diphosphate and organically modified clay act 15 synergistically to improve fire performance. No mention, however, is made of other important aspect such as the effect on mechanical performance, smoke and toxic gas emission.

Takekoshim (US 5,773,502 (1998) General Electric 20 Company) teaches that conventional halogenated-Sb₂O₃ flame-retardant systems and organically modified clay can act synergistically. Takekoshim claims that nano-dispersed clay allows for reductions in the amount of Sb₂O₃ and halogenated flame retardant required to maintain a VO 25 rating in the UL 94 flammability test. Clearly any use of halogenated flame retardant is undesirable.

Masaru, T (JP 10182141 (1998) Sumitoma, Chem Co.) disclose a fire resistant and thermally expandable material at temperatures between 100 to 150°C whereby 30 blowing reagents such as those containing azo, diazo, azide or triazine compound are located between the layers of the silicate. In many polymeric systems, however, this flame retarding system is undesirable since they require moulding or forming at temperatures between 100°C to 150°C.

35 Inoue and Hosokawa (JP 10081510 (1998) Showa Denko K.K.) investigated the use of fluorinated synthetic mica exchanged with melamine (0.1 - 40%) and melamine

- 5 -

salts (<10%) as a means of flame proofing plastics in a two step extrusion process. They claim that a VO rated Nylon6 (UL94 vertical burn test) was achieved at a loading of 5 percent-modified mica when greater than 80% 5 exfoliation occurred. The use of synthetic clays and multiple step processing is clearly undesirable from a commercial viewpoint. Inoue and Hosokawa do not disclose highly desirable chemistries and methodologies associated with triazine based formulations which effect mechanical 10 and fire performance. Furthermore, they do not disclose important methodologies to flame retarded thin parts known by those in the art to be extremely difficult to render flame resistant whilst simultaneously reducing toxic gas and smoke generation during combustion.

15 In a later disclosure Inoue, H., and co-workers (US 6294599 (2001) Showa Denko K.K.) also teach that polyamides reinforced with fibrous additives may be rendered flame resistant through the addition of triazine-modified clay and additional flame retardant. They 20 describe a highly rigid flame-retardant polyamide comprising a polyamide, silicate - triazine compound reinforcement and flame retardant/adjunct. The poor rheological properties of highly rigid polyamide 25 formulations limit the inventions usefulness in preparing components made by conventional processing techniques such as rotational or blow moulding, that are complex or thin walled or which require high ductility or impact performance.

30 Brown, S.C. et al (WO 00/66657, Alcan International) disclose a polymeric material incorporating Cloisite montmorillonite in combination with $\text{Al}(\text{OH})_3$ for the production of fire resistant cables. This strategy is clearly only suitable for plastics that are processed at 35 low temperatures considering that $\text{Al}(\text{OH})_3$ decomposes to release water vapor at temperatures above approximately 190°C.

- 6 -

Accordingly, there is a need for the development of new flame retarding systems which both meet the performance criteria associated with specific applications and address the above concerns.

5

SUMMARY OF THE INVENTION

According to one aspect of the present invention there is provided an inorganic-organic hybrid (IOH) which comprises:

10

(i) an expandable or swellable layered inorganic component; and

(ii) an organic component including at least one ionic organic component.

15

Preferably, the organic component of the IOH also includes one or more neutral organic components which are intercalated between and/or associated with the layer(s) of the inorganic component.

20

According to another aspect of the present invention there is provided a method for the preparation of the IOH defined above which comprises mixing components (i) and (ii) defined above or constituents thereof in one or more steps.

25

The present invention also provides the use of the IOH defined above as a fire resistant material.

30

According to a further aspect of the present invention there is provided a fire resistant formulation which comprises:

- (i) the IOH defined above; and
- (ii) one or more flame retardants.

35

According to a still further aspect of the present invention there is provided a method for the preparation of the fire resistant formulation defined above which comprises mixing components (i) and (ii) as defined above or constituents thereof in one or more steps.

The present invention also provides a polyamide fire resistant formulation which comprises either:

- 7 -

The present invention further provides a method for the preparation of the polyamide fire resistant formulation defined above which comprises dispersing the IOH or the fire resistant formulation defined above or 10 constituents thereof into the polyamide based matrix in one or more steps.

The IOP and/or fire resistant formulations of the present invention may be used to produce fire resistant articles or parts thereof.

15 Thus, the present invention provides a fire
resistant article or parts thereof which is composed
wholly or partly of the IOH and/or fire resistant
formulations defined above.

The present invention also provides a method of
20 preparing the fire resistant article or parts thereof
defined above which comprises moulding or forming the IOH
and/or fire resistant formulations defined above.

DETAILED DESCRIPTION OF THE INVENTION

25 For the purposes of this specification it will
be clearly understood that the word "comprising" means
"including but not limited to", and that the word
"comprises" has a corresponding meaning. It should also be
noted that for the purposes of this specification the
30 terms "swellable" and "expandable" relating to the layered
inorganic component are interchangeable.

The inorganic component is a swellable/expandable layered inorganic based material, rendered positively (or negatively) charged due to isomorphic substitution of elements within the layers, such as, those based on a 1:1 layered silicate structure such as kaolin and serpentine and a 2:1 layered silicate

- 8 -

structure such as phyllosilicates, talc and pyrophyllite. Other useful layered minerals include layered double hydroxides of the general formula $Mg_6Al_{3.4}(OH)_{18.8}(CO_3)_{1.7} \cdot H_2O$ including hydrotalcites and synthetically prepared layered materials including synthetic hectorite, montmorillonite, fluorinated synthetic mica and synthetic hydrotalcite.

5 The group consisting of naturally occurring or synthetic analogues of phyllosilicates is particularly preferred. This group includes smectite clays such as 10 montmorillonite, nontronite, beidellite, volkonskoite, hectorite, bentonite, saponite, sauconite, magadiite, kanyaite, laponite, vermiculite, synthetic micromica (Somasif) and synthetic hectorite (Lucentite). Other 15 useful layered minerals include illite minerals such as ledikite and mixtures of illite minerals with said clay minerals.

Naturally occurring phyllosilicates such as bentonite, montmorillonite, and hectorite are most preferred. Such phyllosilicates with platelet thicknesses 20 less than about 5 nanometers and aspect ratios greater than about 10:1, more preferably greater than about 50:1 and most preferably greater than about 100:1 are particularly useful.

The preferred inorganic materials generally 25 include interlayer or exchangable metal cations to balance the charge, such as, alkali metals or alkali earth metals, for example, Na^+ , K^+ , Mg^{2+} or Ca^{2+} , preferably Na^+ . The cation exchange capacity of the inorganic material should preferably be less than about 400 milli-equivalents per 30 100 grams, most preferably about 50 to about 200 milli-equivalents per 100 grams.

The organic component includes one or more ionic species that may be exchanged with the exchangable metal ions associated with the inorganic component and 35 optionally one or more neutral organic species which are intercalated between and/or associated with the layer(s)

- 9 -

of the inorganic component and/or one or more coupling reagents.

The term "associated with" is used herein in its broadest sense and refers to the neutral organic component being attached to the layer(s) of the inorganic component, for example, by secondary bonding interactions, such as, Van der Waals interactions or hydrogen bonding or trapped by steric limitation.

Suitable examples of ionic species include those that contain onium ions such as ammonium (primary, secondary, tertiary and quaternary), phosphonium or sulfonium derivatives of aliphatic, aromatic or aryl-aliphatic amines, phosphines and sulfides.

Such compounds may be prepared by any method known to those skilled in the art. For example, salts prepared by acid-base type reactions with mineral or organic acids including hydrochloric, sulfuric, nitric, phosphoric, acetic and formic acids, by Lewis-acid - Lewis-base type reactions or by reaction with alkyl halides to form quaternary salts for example using Menschutkin type methodology.

Ionic or neutral compounds which are known to decompose or sublime endothermically, and/or which release volatiles with low combustibility on decomposition and/or induce charring of organic species during thermal decomposition or combustion are particularly preferred.

Suitable species include neutral or ionic derivatives of nitrogen based molecules, such as, triazine based species, for example, melamine, triphenyl melamine, melam (1,3,5-triazine-2,4,6-triamine-n-(4,6-diamino-1,3,5-triazine-yl)), melem ((-2,5,8-triamino-1,3,4,6,7,9,9b-heptaazaphenalenene)), melon (poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalenene-2,5-diyl)imino}), bis and triaziridinyltriazine, trimethylsilyltriazine, melamine cyanurate, melamine phthalate, melamine phosphate, melamine phosphite, melamine phthalimide, dimelamine phosphate, phosphazines and/or low molecular weight

- 10 -

polymers with triazine and phosphazine repeat units or salts or derivatives of the above molecules including onium ion derivatives or salts or derivatives of isocyanuric acid, such as, isocyanuric acid, cyanuric acid, triethyl cyanurate, melamine cyanurate, triglycidylcyanurate, triallyl isocyanurate, trichloroisocyanuric acid, 1,3,5-tris(2-hydroxyethyl)triazine-2,4,6-trione, hexamethylenetetramine, melam cyanurate, melam cyanurate and melon cyanurate.

Reagents known to induce charring of organic species include derivatives of phosphoric acid or boric acid, such as ammonia polyphosphate and melamine polyphosphate, melamine phosphate ammonium borate.

In another embodiment of the invention, the preferred ionic compounds may be optionally used in combination with other ionic compounds, for example, those known to improve compatibility and dispersion between the layered inorganic material and polymeric matrices such as those described in WO 93/04118 for the preparation of nanomaterials. Amphiphilic molecules that incorporate a hydrophilic ionic group along with hydrophobic alkyl or aromatic moieties are preferred.

One or more coupling reagents may also be associated with the inorganic component. Suitable coupling reagents include organically functionalised silanes, zirconates and titanates. Examples of silane coupling reagents include tri-alkoxy, acetoxy and halosilanes functionalised with amino, epoxy, isocyanate, hydroxyl, thiol, mercapto and/or methacryl reactive moieties or modified to incorporate functional groups based on triazine derivatives, long chain alkyl, aromatic or alkylaromatic moieties. Examples of zirconate and titanate coupling reagents include Teaz and Titan1.

It is known in the art that metal cations or anions associated with layered inorganic materials may be exchanged with organic ions through ion exchange

- 11 -

processes. In a typical process, the layered inorganic material is first swollen or expanded in a suitable solvent(s) prior to ion exchange and then collected from the swelling solvent following agglomeration using methods 5 such as filtration, centrifugation, evaporation or sublimation of the solvent. Ion exchange techniques with suitable molecules are known to be a useful method of increasing the compatibility between clay and organic polymeric binders; thus aiding dispersion of clay 10 platelets into polymeric based matrices on a nanometer scale.

We have discovered that the ion exchange process may be optionally carried out in the presence of one or more types of organic ion to produce an inorganic-organic 15 hybrid with a plurality of functions. Without wishing to limit the present invention, such functions may include the presence of ions which promote dispersion, compatibility and interactions with the plastic matrix and ions useful to improve other properties such as fire 20 performance. Generally during ion exchange the organic ions are added in molar excess of the ion exchange capacity of the inorganic material, preferably less than about 10-fold excess, more preferably less than about a 5-fold excess is required.

25 It has also been unexpectedly discovered that the ion exchange processes may be carried out in the presence of functional dissolved or partially dissolved neutral species. Without being limited by theory, it is proposed that at least a portion of the neutral species 30 are trapped in the intergallery region or otherwise associated with the layered inorganic material following ion exchange. Such a process provides a useful mechanism of dispersing neutral additives on a molecular level into plastics. Again without being limited by theory, during 35 melt processing at least partial exfoliation of the inorganic-organic hybrid allows the neutral molecules to diffuse away and become homogeneously dispersed with the

- 12 -

matrix on a molecular level. This has a major impact on the performance of the resultant material since it is well known that efficient dispersion of all components in a plastic formulation, preferably on a nano- or molecular scale, is an important factor for achieving optimum performance.

In another aspect of the invention, the IOH may be treated prior, during or following ion exchange with one or more coupling reagents as described above. The coupling reagents are derivatized to improve, for example, the compatibility and interactions between the inorganic phase and polymeric matrix or to attach other desirable functionalities to the inorganic layered phase.

Suitable flame retardants which retard flame propagation, heat release and/or smoke generation which may be added singularly or optionally synergistically to the IOH include:

- Phosphorus derivatives such as molecules containing phosphate, polyphosphate, phosphites, phosphazine and phosphine functional groups, for example, melamine phosphate, dimelamine phosphate, melamine polyphosphate, ammonia phosphate, ammonia polyphosphate, pentaerythritol phosphate, melamine phosphite and triphenyl phosphine.
- Nitrogen containing derivatives such as melamine, melamine cyanurate, melamine phthalate, melamine phthalimide, melam, melem, melon, melam cyanurate, melem cyanurate, melon cyanurate, hexamethylene tetraamine, imidazole, adenine, guanine, cytosine and thymine.
- Molecules containing borate functional groups such as ammonia borate and zinc borate.
- Molecules containing two or more alcohol groups such as pentaerythritol, polyethylene alcohol, polyglycols and carbohydrates, for example, glucose, sucrose and starch.
- Molecules which endothermically release non-combustible decomposition gases, such as, metal

- 13 -

hydroxides, for example, magnesium hydroxide and aluminum hydroxide.

- Expandable graphite

The polyamide based matrix may be included in
5 the fire resistant formulation in pellet, granule, flake
or powdered form. Suitable polyamides comprise generic
groups with repeat units based on amides, such as, Nylon4,
Nylon6, Nylon7, Nylon 11 and Nylon12, Nylon46, Nylon66,
Nylon 68, Nylon610, Nylon612 and aromatic polyamides, for
10 example, poly'm'phenyleneisophthalamide and
poly'p'phenylene'terephthalamide.

It will be appreciated that the polyamide based matrix may include co-polymers, blends and alloys. The co-polymers may be made up of two or more different repeat units one of which is an amide. Such co-polymers may be prepared by any suitable methods known in the art, for example, at the point of initial polymerisation or later through grafting or chain extension type reactions during processing. The polyamide blends and alloys may be prepared using any method known to those skilled in the art including melt or solution blending. Blending or alloying the polyamide with other polymers may be desirable to improve properties such as toughness, modulus, strength, creep, durability, thermal resistance, conductivity or fire performance.

Nylon12, Nylon6 and Nylon66 and their respective co-polymers, alloys and blends are particularly preferred.

The polyamide formulation can also optionally contain one or more additives known in the art of polymer processing, such as, polymeric stabilisers, for example, UV, light and thermal stabilisers; lubricants; antioxidants; pigments, dyes or other additives to alter the materials optical properties or colour; conductive fillers or fibers; release agents; slip agents; plasticisers; antibacterial or fungal agents, and processing agents, for example, dispersing reagents, foaming or blowing agents, surfactants, waxes, coupling

- 14 -

reagents, rheology modifiers, film forming reagents and free radical generating reagents.

A particularly preferred formulation comprises Nylon12, Nylon6 and/or Nylon66; montmorillonite modified with melamine hydrochloride and/or melamine; melamine cyanurate and/or melam (1,3,5-triazine-2,4,6-triamine-n-(4,6-diamino-1,3,5-triazine-yl)) cyanurate, and/or melem ((-2,5,8-triamino-1,3,4,6,7,9,9b-heptaazaphenalene)) cyanurate and/or melon (poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalene-2,5-diyl)imino}) cyanurate; magnesium hydroxide; and one or more additives.

The polyamide formulation preferably contains a polyamide based matrix in an amount of from about 50 to about 95% w/w, an IOH in an amount less than about 25% w/w and optionally a flame retardant and/or additives in an amount less than about 30% w/w, but in some cases preferably above about 10% w/w.

It has been discovered that the IOH may be readily dispersed into the polyamide based matrix during the compounding (mixing) stage. Without wishing to be limited by theory, it is proposed that ion exchange enhances the layered IOHs compatibility with polyamides compared with unmodified inorganic layered materials. This heightened compatibility in combination with sufficient mixing forces, appropriate mixing sequence, screw design and time allows the organically modified platelets associated with the IOH to be at least partially exfoliated into the polyamide and hence dispersed at least partially on a nanometer scale. This process also provides a useful mechanism of dispersing into the polyamide any neutral molecules associated with the IOH on a molecular level.

Dispersion of the various components of the fire resistant formulation including the IOH is aided by grinding prior to mixing. Grinding is achieved using any suitable grinding equipment including ball mills, ring mills and the like. It is preferable that the components

- 15 -

including the IOH is ground to a particle size less than about 200 microns, more preferably less than about 50 microns, most preferably less than about 20 microns. The hybrid material may also be ground using specialty 5 grinding equipment allowing grinding to nanometer sizes.

Dispersion may be affected using any suitable melt, solution or powder based mixing process allowing sufficient shear rate, shear stress and residence time to disperse the IOH at least partially on a nanometer scale. 10 Such processes may be conducted using milling procedures such as ball milling, in a batch mixer using internal mixers, such as, Banbury and Brabender/Haake type mixers, kneaders, such as, BUS kneaders, continuous mixing processes including continuous compounders, high intensity 15 single and twin screw extrusion.

Melt processing is preferred and in a particularly preferred embodiment, twin screw extruders with an L:D ratio of at least about 24, preferably more than about 30 equipped with at least one and preferably 20 multiple mixing and venting zones are employed for dispersion. Such screw configurations useful for dispersive and distributive mixing are well known to those in the art. A particularly useful system has been found to be that illustrated in Figure 1.

25 The components of the formulation may be added in any order or at any point along the extruder barrel. Since polyamides are susceptible to hydrolysis it is preferable that the components are dried prior to processing and/or mechanisms to remove water vapor such as 30 vents or vacuum ports available during processing. In a preferred embodiment, all of the components are added at one end of the extruder. In another preferred embodiment, a polymeric binder and optionally minor components are added at one end of the extruder and the IOH and 35 optionally minor components at a later point/s. In still another preferred embodiment, the IOH portion of the polymeric binder and optionally minor components are added

- 16 -

at one end of the extruder with the remaining portion of the polymeric binder and optionally minor components are added at a later point/s. Following extrusion the molten composition is cooled by means of water bath, air knife or 5 atmospheric cooling and optionally cut into pellets.

Preferably all of the major and minor components of the system can be combined in as few a mixing steps as possible, most preferably in a single mixing step.

The moulding or forming of the polyamide 10 formulation into fire resistant articles or parts thereof can be carried out using any method known to those in the art including processes such as extrusion, injection moulding, compression moulding, rotational moulding, blow moulding, sintering, thermoforming, calendering or 15 combinations thereof.

In one embodiment of the invention the fire 20 resistant polyamide system containing the major and minor components is moulded or formed into parts having wall thickness less than about 25 mm, preferably less than about 5 mm, most preferably less than 1.5 mm. Such parts include but are not limited to tubes, complex moulded hollow parts, sheets and complex moulded sheets and other complex objects that are moulded or formed using 25 techniques, such as, extrusion, injection moulding thermoforming and rotational moulding.

In the simplest process, the article or part is directly produced during compounding for example by locating a die at the end of the extruder allowing the shape of the extrudate to be modified as required. 30 Examples of such components include simple parts such as film, tape, sheet, tube, rod or string shapes. The process may also involve multiple layers of different materials one of which being the said polymeric system built up by processes known to those in the art including 35 co-extrusion.

In another preferred embodiment, the formulation is moulded or formed in a separate step using techniques

- 17 -

such as injection, compression or blow moulding. Such parts are generally more complex in nature compared with parts formed by extrusion alone, their design only limited by the requirements of the moulding tool / process 5 employed. Suitable examples include but are not limited to stowage bin hinge covers, ECS duct spuds, latches, brackets, passenger surface units and the like.

It is noted that for certain applications it may be preferable that the fire resistant polyamide 10 formulation is ground to a powder. In such cases it has unexpectedly been found that grinding of the said formulation using cryogenic or atmospheric grinding techniques known to those in the art may be carried out without significantly effecting the performance of the 15 system. Such moulding applications include selective laser sintering, rotational moulding, and extrusion. Suitable examples including but not limited to environmental control systems (air-conditioning ducts) and the like.

20 In other preferred applications, the polymeric formulation may be first formed into a sheet or film, for example, through extrusion, blow moulding, compression moulding or calendering. The sheet may be subsequently moulded to a desired shape using thermoforming techniques. 25 In yet another application, the sheet or film may be used to prepare reinforced thermoplastic laminates with woven fabrics prepared from surface modified or natural glass, carbon or aramid using techniques such as compression moulding or resin infusion/transfer. Again, the laminate 30 sheet hence formed may be further moulded to a desired shape using techniques such as thermoforming.

Alternatively the formulation may be spun into fibres by any method known to those skilled in the art. Such a process provides a method for producing fire 35 resistant fabrics, carpets and alike

The present invention is useful for producing polyamide materials with favourable rheological properties

- 18 -

for moulding including thin or intricate articles or parts thereof which maintain mechanical properties close to or exceeding that of the virgin polyamide matrix and which show improved fire performance in standard tests through 5 resisting combustion by self-extinguishing when ignited, limiting flame propagation, and generating low smoke and toxic gas emissions. Such articles or parts thereof are useful for applications which require superior fire performance and in industries that are regulated for fire 10 performance including transport, for example, air, automotive, aerospace and nautical; building and construction; and electrical or optical, for example, cables, wires and fibres.

15 BRIEF DESCRIPTION OF THE DRAWINGS

In the examples, reference will be made to the accompanying drawings in which:

Figure 1 is a diagram showing the twin screw extruder screw and barrel configuration;
20 Figure 2 is a graph showing the XRD results and transmission electron microscope (TEM) image for Example 7;

Figure 3 is a graph showing the XRD results for Example 8;
25 Figure 4 is a graph showing the XRD results for Example 9;

Figure 5 is a graph showing XRD results for Example 17; and

30 Figure 6 is a picture of complex hollow fire resistant components moulded with formulations 13 and 34.

EXAMPLES

The invention will now be described with reference to the following non-limiting examples.

35 General Conditions & Reagents

- 19 -

Tables 1, 2 and 3 Outline General Reagents,
Conditions & Procedures associated with the examples.

- 20 -

Table 1: Commercially Available Reagents

Reagent	Trade name	Supplier
Montmorillonite – organic modified	Cloisite 93A	Southern Clay
Montmorillonite – organic modified	Cloisite 30B	Southern Clay
Montmorillonite	Cloisite Na ⁺	Southern Clay
Synthetic Hectorite	Laponite	Southern Clay
Nylon12	Vestamid 9005	Degussa
Nylon12 FR (Flame retarded)	Vestamid 7166	Degussa
Polyetherimide	Ultem 9075	GE Plastics
Nylon6	Akulon PA6	DSM
Nylon66	Akulon PA66	DSM
Cyanuric acid	Cyanuric acid	Aldrich
Melamine cyanurate	Fyrol MC	Akzo-Nobel
Melamine phosphate	Fyrol MP	Akzo-Nobel
Melamine polyphosphate	Melapur 200	DSM Melapur
Melamine	Melamine	Aldrich
Pentaerythritol	Pentaerythritol	Aldrich
Magnesium hydroxide	Magnifin	Martinswerk
Ammonia polyphosphate	Antiblaze MC	Rhodia
Pentaerythritol phosphate	NH-1197	Great Lakes
Pentaerythritol phosphate Blend	NH-1511	Great Lakes
Zinc borate	Fire Brake ZB	US Borax
Zn Stearate	Zincum	Baerlocher
Ca Stearate	Ceasit	Baerlocher
Int 38	Synthetic resin	AXEL
LuWax Eas1	Ethylene co-polymer	BASF
Irganox b1171	Phosphite/hindered phenol blend	CIBA

- 21 -

Table 2: Processing Equipment and Conditions

Equipment	Type																																				
Twin screw extruder	<p>Berstorff ZE 25 mm modular co-rotating twin screw extruder coupled to a Haake Rheocord motor drive and torque cell for rheology measurement</p> <p>L:D ratio = 36:1</p> <p>Screw and barrel configuration presented in Figure 1,</p> <p>Screw speed 300 rpm</p> <p>Feed rate ~1.2 Kg/hour</p> <p>Residence time average 2 min</p> <p>Flat 200°C temperature profile from throat to die (nylon12)</p> <p>Flat 250°C temperature profile from throat to die (nylon6)</p> <p>Flat 275°C temperature profile from throat to die (nylon66)</p>																																				
Batch Mixer	<p>Haake R3000 batch mixer connected to torque rheological load cell, pneumatic ram, roller rotors</p> <p>Rotor speed – 5 min 60 rpm, 10 min 120 rpm</p> <p>Temperature 190°C</p>																																				
Injection Moulding	<p>Battenfeld 80 ton BA 800 CDC injection moulding machine</p> <p>Temperature profile:</p> <table> <tr> <td>Nylon12</td> <td>Zone 1</td> <td>2</td> <td>3</td> <td>Nozzle</td> <td>Die</td> </tr> <tr> <td>Temp (°C)</td> <td>215</td> <td>220</td> <td>225</td> <td>225</td> <td>70°C</td> </tr> </table> <table> <tr> <td>Nylon6</td> <td>Zone 1</td> <td>2</td> <td>3</td> <td>Nozzle</td> <td>Die</td> </tr> <tr> <td>Temp (°C)</td> <td>230</td> <td>230</td> <td>250</td> <td>260</td> <td>90°C</td> </tr> </table> <table> <tr> <td>Nylon 66</td> <td>Zone 1</td> <td>2</td> <td>3</td> <td>Nozzle</td> <td>Die</td> </tr> <tr> <td>Temp (°C)</td> <td>260</td> <td>260</td> <td>280</td> <td>290</td> <td>90°C</td> </tr> </table> <p>ASTM test samples:</p> <p>Injection pressure gradient 800 to 600 bar, cavity pressure 400 bar,</p> <p>Holding pressures 600 to 0 bar</p> <p>Cooling time 30sec</p> <p>Cone Calorimetry Samples:</p> <p>Injection pressure gradient 950 to 650 bar, cavity pressure 325 bar,</p> <p>Holding pressures 650 to 0 bar</p> <p>Cooling time 60 sec</p>	Nylon12	Zone 1	2	3	Nozzle	Die	Temp (°C)	215	220	225	225	70°C	Nylon6	Zone 1	2	3	Nozzle	Die	Temp (°C)	230	230	250	260	90°C	Nylon 66	Zone 1	2	3	Nozzle	Die	Temp (°C)	260	260	280	290	90°C
Nylon12	Zone 1	2	3	Nozzle	Die																																
Temp (°C)	215	220	225	225	70°C																																
Nylon6	Zone 1	2	3	Nozzle	Die																																
Temp (°C)	230	230	250	260	90°C																																
Nylon 66	Zone 1	2	3	Nozzle	Die																																
Temp (°C)	260	260	280	290	90°C																																

- 22 -

Table 2 cont

Equipment	Type
Compression Moulding	Assett 2.5 MPa pneumatic press, 45 cm platens, heating (400°C) and cooling Moulding platen temperature 220°C nylon12 Moulding platen temperature 260°C nylon6 Moulding platen temperature 290°C nylon66

Table 3: Characterization Techniques, Conditions and Sample Preparations

Equipment	Type
X-ray diffraction (XRD)	Phillips PW 1729, CuK _{α1} source $\lambda = 0.154$ nm Powders were ground to a particle size of less than 100 micron, Plastics were compression moulded (210°C) to a thickness of 100 micron
Transmission Electron Microscopy (TEM)	Hitachi H-7500 operating at an electron potential of 120 kV 100 nm thick sections were prepared by ultra microtomy
Differential Scanning Calorimetry (DSC)	Cryogenic TA 2920 MDSC employing Advantage software, 10°C and 20°C/min ramp rate rates for heating and cooling for general thermal and glass transition respectively. Calibrated against, Indium, distilled water, cyclohexane and sapphire Powders were ground to a particle size of less than 100 micron. Plastics were compression moulded (210°C) to a thickness of 100 micron with quench cooling, 5mm diameter specimens were punched from the moulded sheet
Thermal Gravimetric Analysis (TGA)	Thermal Sciences, PL-STA, referenced against Al ₂ O ₃ Heating rate ramp 10°C / min Powders were ground to a particle size of less than 100 micron Plastics were compression moulded (210°C) to a thickness of 100 micron with quench cooling, 4mm diameter specimens were punched from the moulded sheet
Cone Calorimetry Testing	ASTM E 1354 - 92 Modified from the original Stanton-Redcroft model, employing CSIRO developed software Radiant flux 35 kW/m ² , 3 repeats per sample, ASTM E1356 Following injection moulding, samples (100x100x6mm) were conditioned for 7 days at 23°C at 50% RH. Heat release, smoke, mass loss and gas emission were measured
Radiant Panel	Conducted as per FAA specification (DOT FAA/AR-0012) & as outlined in ASTM E648 – 93a

Table 3 cont.

Equipment	Type
Specific Optical Density of smoke Generated By combustion Solid Materials and gas emission	ASTM E662 – 93 for optical density with gas released by samples during the test analyzed for HF, HCl, HCN, H ₂ S, NO _x , HBr, PO ₄ , SO ₂
Vertical Burn	<p>Vertical burn tests according to UL94 or FAA specifications.</p> <p>UL94 specification –</p> <p>One 10 sec application of flame from a 10 mm burner to 125x12.3x3.2 mm samples according to UL specifications 2000.</p> <p>Flame extinguish times were monitored over at least 3 samples</p> <p>Extinguishing times, VO <10s, V1 <30s, V2 <30s</p> <p>Cotton Wool Ignition No No Yes</p> <p>FAA (DOT FAA/AR-0012) and ASTM F501-93</p> <p>12 s burn</p> <p>One 12s application of flame from a 10 mm burner to 300x75 mm samples according to FAA specification 2000: sample thickness specified</p> <p>Pass FAA test requirement:</p> <p>Flame extinguished <15sec</p> <p>Drip extinguished <5 sec</p> <p>Burn height <203 mm</p> <p>60 s burn</p> <p>One 60s application of flame from a 10 mm burner to 300x75 mm samples according to FAA specification 2000</p> <p>Pass FAA test requirement:</p> <p>Flame extinguished <15sec</p> <p>Drip extinguished <3 sec</p> <p>Burn height <150 mm</p> <p>Sample thickness specified</p>

- 25 -

Table 3 cont.

Equipment	Type
IZOD Notched Impact Testing	Radman ITR 2000 instrumented impact tester Izod mode, impact strain rate 3.5 ± 0.2 m/sec 10 repeats per sample, ASTM 256 Following injection moulding, samples were stored for 24 h in desiccated containers, notched according to the ASTM 256 standard and tested 'dry as moulded standard deviation generally less than 8%
Tensile Testing	Instron tensile testing apparatus (5565) utilizing a 30 kN load cell, 50 mm/min strain rate 5 repeats per sample as per ASTM D638 External extensometer used for independent modulus measurements ASTM D5938 Following injection moulding, samples were stored for 24 h in desiccated containers and tested 'dry as moulded' Generally standard deviation less than 2% for modulus and strength results
MFI	MFI testing was completed according to ASTM D1238 standards employing 2.16 load at a temperature of 235°C, Employing a Davenport Melt Flow Indexer apparatus
Parallel Plate Rheology	The viscosities of samples were measured over a wide range of shear rate range of 10^{-2} to 10^1 s ⁻¹ at 240 °C. Tests of shear rate sweep were carried out using a shear strain-controlled rheometer, RDA II (Rheometric Scientific Inc.). The test fixture geometry used was 25 mm parallel-plate with a constant gap between 0.6 – 0.8 mm. The nitrogen gas was used to provide an inert testing environment to reduce sample degradation due to oxidation of samples.

Methods for Preparing Inorganic-Organic hybrids (IOH) -
Examples 1-6

Example 1: Preparation of melamine hydrochloride
modified montmorillonite (IOH1)

Montmorillonite exchanged Na^+ (Cation Exchange Capacity (CEC) = 92 meq/100g) was suspended in 80°C DI water (2% w/w) and mechanically stirred at 1500 rpm for 60 min.

10 Melamine monohydrochloride salt (1.4 mmol/100g montmorillonite) was then added to the solution and the resultant suspension allowed to cool with continued stirring for a further 150 min. Following filtration of the suspension, the precipitate was thoroughly washed with warm DI water and then preliminary dried (60 – 80°C). The resultant granular organically modified clay was ground to a particle size of less than 50 micron and then further dried at 75°C prior to processing or analysis.

20 **XRD** (CuK α 1 source $\lambda = 0.154$ nm)

Cation	Na^+	Melamine.HCl modified Montmorillonite
XRD d_{001}	1.10 nm	1.27 nm

Results indicate that with ion exchange montmorillonite's intergallery spacing is increased from 25 1.10 nm to 1.27 nm. This result is consistent with sodium ions being replaced by protonated melamine ions in the intergallery region during ion exchange.

- 27 -

Example 2a: Preparation of melamine hydrochloride modified montmorillonite in the presence of melamine (IOH2)

5 Montmorillonite exchanged Na^+ (Cation Exchange Capacity (CEC) = 92 meq/100g) was suspended in 80°C DI water (2% w/w), melamine added (1.4 mmol/100 g montmorillonite) and the solution mechanically stirred at 1500 rpm for 60 min. Melamine monohydrochloride salt (1.4 mmol/100g montmorillonite) was then added to the solution and the resultant suspension allowed to cool with continued stirring for a further 150 min. Following filtration of the suspension, the precipitate was thoroughly washed with warm DI water and then preliminary dried (60 - 80°C). The 15 resultant granular organically modified clay was ground to a particle size of less than 50 micron and then further dried at 75°C prior to processing or analysis.

XRD (CuK α 1 source $\lambda = 0.154$ nm)

20

Cation	Na^+	Melamine and Melamine.HCl modified montmorillonite
XRD d_{001}	1.10 nm	1.39 nm

Results indicate that montmorillonite modified by melamine hydrochloride in the presence of melamine has an expanded intergallery spacing compared with both 25 montmorillonite that is modified with melamine hydrochloride or sodium ions alone. The result is consistent association/entrapment of the neutral melamine with the clay during ion exchange.

- 28 -

Example 2b: Preparation of melamine hydrochloride modified montmorillonite in the presence of melamine (IOH2)

5 3.0Kg of sodium montmorillonite was dispersed into 200L de-ionized water at 60°C with vigorous stirring (200 rpm) adding the powder slowly over a period of approximately one hour to assist wetting out of the individual particles/platelets. After the suspension had stirred at 10 that temperature for approximately 2 hours, an aqueous solution (35L) containing 1.39 Kg melamine and 0.92L HCl (9.65M) at 85°C was rapidly added whilst the impeller speed was simultaneously increased to 300 rpm. After an initial period of high viscosity whilst the modified 15 montmorillonite aggregated, the viscosity decreased and the clay solution was allowed to stir for a further 3 hours at 60°C. Following filtration of the suspension the collected modified clay was re-dispersed into de-ionized water (150L) and allowed to stir for 1 hour at 60°C before 20 an aqueous solution (10 L) containing 0.385Kg melamine and 0.26 L HCl (9.65M) at approx 85°C was added. At this point the mixture was stirred for a further two hours before it was filtered. Next the modified clay was re-dispersed 25 into de-ionized water (150L) and stirred for a further 1 hour at 60°C prior to filtration, drying and grinding of the modified clay to a particle size less than 50 micron.

XRD (CuK α 1 source $\lambda = 0.154$ nm)

Cation	Na ⁺	Melamine and Melamine.HCl modified Montmorillonite
XRD d₀₀₁	1.10 nm	1.40 nm

30

These results illustrate that the robustness of the modification procedure to variation in mole ratio of montmorillonite CEC to melamine salt and melamine and the reaction conditions employed to carry out the modification

- 29 -

procedure. This result is consistent association/entrapment of the neutral melamine with the clay during ion exchange.

5 Example 2c: Preparation of melamine hydrochloride modified montmorillonite in the presence of melamine (IOH2)

15.0Kg of montmorillonite was dispersed into 200L de-ionized water at 60°C with vigorous stirring (200 rpm) adding the powder slowly over a period of approximately 2 hours to assist wetting out of the individual particles/platelets. After the suspension had stirred at that temperature for approximately 4 hours, an aqueous solution (50L) containing 2.78Kg melamine and 1.84 L HCl (9.65 M) at 85°C was rapidly added whilst the impeller speed was simultaneously increased to 300 rpm. After an initial period of high viscosity whilst the modified montmorillonite aggregated, the viscosity decreased and the clay solution was allowed to stir for a further 3 hours at 60°C. Following filtration of the suspension the collected modified clay was re-dispersed into de-ionized water (150L) and allowed to stir for 1 hour at 60°C before an aqueous solution (25L) containing 1.925 Kg melamine and 1.3 L HCl (9.65M) at approx 85°C was added. At this point the mixture was stirred for a further two hours before it was filtered. Next the modified clay was re-dispersed into de-ionized water (200L) and stirred for a further 1 hour at 60°C prior to filtration, drying and grinding of the modified clay to a particle size less than 50 micron.

XRD (CuK_{α1} source $\lambda = 0.154$ nm)

Cation	Na ⁺	Melamine and Melamine.HCl modified Montmorillonite
XRD d_{001}	1.10 nm	1.40 nm

- 30 -

Results illustrate the robustness of the modification procedure to variation in reaction conditions employed to carry out the modification procedure. This result is consistent with association / entrapment of the neutral melamine molecules with the clay during ion exchange.

5 Example 3: Preparation of melamine cyanurate hydrochloride modified montmorillonite (IOH3)

10 Na^+ exchanged montmorillonite (Cation Exchange Capacity (CEC) = 92 meq/100g) was suspended in 95°C distilled water (2% w/w), cyanuric acid added (1.4 mmol/100g montmorillonite) and the solution mechanically stirred at 1500 rpm for 60 min. Melamine mono-hydrochloride salt (1.4 mmol/100g montmorillonite) was then added to the solution and the resultant suspension with continued stirring for a further 150 min. Following filtration of 15 the suspension, the precipitate was thoroughly washed with warm distilled water and then preliminary dried (75°C). The resultant granular organically modified clay was ground to a particle size of less than 45 micron and then further dried at 60 - 80°C prior to processing or analysis.

20

25 XRD (CuK α 1 source $\lambda = 0.154$ nm)

Cation	Na^+	Melamine cyanurate.HCl modified montmorillonite
XRD d_{001}	1.10 nm	1.42 nm

30 Results from Example 3 indicate that the intergallery spacing of montmorillonite is expanded further when exchanged with melamine cyanurate ion compared with sodium ion or melamine ion modified

- 31 -

montmorillonite alone (Example 1) due to its larger size and hence steric impact.

5 Example 4: Preparation of melamine and melamine cyanurate modified montmorillonite in presence of melamine and melamine cyanurate (IOH4)

10 Montmorillonite exchanged Na^+ (Cation Exchange Capacity (CEC) = 92 meq/100g) was suspended in 95°C distilled water (2% w/w), cyanuric acid added (1.4 mmol/100 g montmorillonite) and the solution mechanically stirred at 1500 rpm for 60 min. Melamine monohydrochloride salt (1.4 mmol/100g montmorillonite) and melamine (1.4 mmol/100g montmorillonite) was then added to the solution and the resultant suspension continued stirring for a further 150 min. Following filtration of the suspension, the precipitate was thoroughly washed with warm distilled water and then preliminary dried under vacuum (75°C). The 20 resultant granular organically modified clay was ground to a particle size of less than 45 micron and then further dried at 60 - 80°C prior to processing or analysis.

25 XRD (CuK α 1 source $\lambda = 0.154$ nm)

Cation	Na^+	Melamine and Melamine cyanurate.HCl modified montmorillonite
XRD d_{001}	1.10 nm	1.53 nm

30 The results from Example 4 indicate that the intergallery spacing of montmorillonite exchanged with melamine cyanurate ion in the presence of melamine and melamine cyanurate is larger than both sodium ion or melamine cyanurate ion exchanged montmorillonite alone (Example 3). This result is consistent with

- 32 -

association/entrapment of the neutral melamine and melamine cyanurate with the clay during ion exchange.

Example 5: Preparation of melamine and trimethyl
cetyl ammonium and melamine hydrochloride
modified montmorillonite (IOH5)

Montmorillonite exchanged Na⁺ (Cation Exchange Capacity (CEC) = 92 meq/100g) was suspended in 90°C distilled water (2% w/w), and the solution mechanically stirred at 1500 rpm for 60 min. Melamine monohydrochloride salt (1.4 mmol/100g montmorillonite) and trimethylcetylammonium chloride (1.4 mmol/100g montmorillonite) was then added to the solution and the resultant suspension allowed to cool with continued stirring for a further 150 min. Following filtration of the suspension, the precipitate was thoroughly washed with warm distilled water and then preliminary dried under vacuum (75°C). The resultant granular organically modified clay was ground to a particle size of less than 45 micron and then further dried at 60 - 80°C prior to processing or analysis.

XRD (CuK α 1 source $\lambda = 0.154$ nm)

Cation	XRD d_{001}
Na^+	1.10 nm
Trimethylcetylammonium chloride	1.84 nm
Melamine and Trimethylcetylammonium chloride modified montmorillonite	1.68 nm

25

The results from Example 5 indicate that the intergallery spacing of montmorillonite exchanged with both trimethylcetylammmonium chloride and melamine hydrochloride is larger than sodium but smaller than trimethylcetylammmonium ion exchanged montmorillonite. This result is consistent with trimethylcetylammmonium chloride

- 33 -

and melamine hydrochloride being present in the intergallery spacing of the modified montmorillonite.

5 Example 6: Preparation of melamine and melamine hydrochloride modified synthetic hectorite, laponite (IOH6)

Hectorite clay (Synthetic Laponite RD) was modified using the same general procedure as employed in Example 2 taking 10 into consideration its lower cation exchange capacity (CEC) of 55 mmol/100g and employing a 1% solution for modification. Strict control was placed over the mole ratio of hectorite CEC and melamine salt to encourage platelet agglomeration. Following treatment with the 15 melamine salt/melamine, the modified synthetic clay was separated from the treatment solution by filtration.

XRD (CuK_{α1} source $\lambda = 0.154$ nm)

Cation	Na ⁺ /Li ⁺	Melamine and Melamine.HCl Modified Hectorite
XRD d_{001}	1.20 nm	1.33 nm

20

The results from Example 6 indicate that the intergallery spacing of synthetic hectorite exchanged with melamine hydrochloride in the presence of melamine is larger than sodium changed montmorillonite.

25

Melt Dispersion of Components and Formulation of Fire resistant Materials Examples 7-20

While each of the following examples use Nylon12, Nylon6 or Nylon66 as the polyamide based matrix, 30 the person skilled in the art will appreciate that the examples for fire retarding nylon12, nylon6 and nylon66 are also applicable to other types of polyamides, polyamide co-polymers, polyamide blends, alloys and the like.

- 34 -

The formulation constituents employed in Examples 7 to 20 are provided in Tables 4a to 4e.

Table 4a Formulations used in Examples 7 to 20

Formulation	Nylon12	Cloisite Na ⁺	Cloisite 30B	Cloisite 93A	IOH2 (Example2)	Melamine Cyanurate
1	99.25		0.75			
2	98.5		1.5			
3	95		5.0			
4	93		7.0			
5	95			5		
6	95		5			
7	82		3			15
8	83.5		1.5			15
9	84.25		0.75			15
10	85					15
11	82				3	15
12	83.5				1.5	15
13	84.25				0.75	15
14	84.5				3	12.5
15	86				1.5	12.5
16	86.75				0.75	12.5
17	87				3	10
18	88.5				1.5	10
19	89.25				0.75	10
20	90.5				3	7.5
21	91				1.5	7.5
22	91.75				0.75	7.5

Table 4b Formulations used in Examples 7 to 20

Formulation	Nylon12	IOH2 (Example 2)	Melamine Cyanurate	Magnesium Hydroxide (H7)	Melamine phosphate	Melamine poly phosphate	Ammonia poly phosphate	Pentaerythritol phosphate blend
23	83.5	1.5		15				
24	83.5	1.5		15				
25	83.5	1.5		15				
26	83.5	1.5		15				
27	83.5	1.5				15		
28	83.5	1.5					15	
29	83.5	1.5						15
30	83.5	1.5		10	5			
31	87.5				12.5			
32	98.5	1.5						

Table 4c Formulations used in Examples 7 to 20

Formulation	Nylon12	IOH2 (Example 2)	Melamine cyanurate	Magnesium hydroxide (H7)	Magnesium hydroxide (H10)	Magnesium hydroxide (H5iv)	Magnesium hydroxide (H10iv)
33	82	3		12.5	2.5		
34	83.5	1.5		12.5	2.5		
35	84.25	0.75		12.5	2.5		
36	82	3		10	5		
37	84.25	0.75		10	5		
38	82	3		7.5	7.5		
39	83.5	1.5		7.5	7.5		
40	84.25	0.75		7.5	7.5		
41	83.5	1.5		12.5		2.5	
42	83.5	1.5		12.5		2.5	
43	83.5	1.5		12.5			2.5

Table 4d
Formulations used in Examples 7 to 20

Formulation	Nylon12	Nylon6	Nylon66	IOH1 (Example 1)	IOH2 (Example 2)	IOH4 (Example 4)	IOH5 (Example 5)	Melamine cyanurate
44	88.5			1.5				10
45	83.5			1.5				15
46	88.5					1.5		10
47	83.5					1.5		15
48		84.25			0.75			15
49			84.25		0.75			15
50		84.25					0.75	15

Table 4e Formulations used in Examples 7 to 20

Formulation	Nylon12	IOH2 (Example 2)	Melamine cyanurate	Calcium stearate	Zinc Stearate	Int38	Luwax EAS1	Irganox
51	83.25	0.75	1.5	1				
52	82.25	0.75	1.5	2				
53	83.25	0.75	1.5		1			
54	82.25	0.75	1.5		2			
55	82.25	0.75	1.5			2		
56	82.25	0.75	1.5				2	
57	83.75	0.75	1.5					0.5

- 40 -

Example 7: Processing rheology (Table 5), XRD & TEM (Figure 2), mechanical (Table 6) and fire performance (Tables 7 & 8) of nylon12 modified with commercially available clay during melt processing.

5

The following example indicates that the processing rheology of Nylon 12 is not affected by the melt dispersion of commercially available 'organoclay' at least partially on a nanometer scale (XRD). This dispersion results in improved mechanical performance and heat release rate as determined by cone calorimetry but poor performance compared with conventional flame retarded nylon 12(Nylon12 FR) in terms of vertical burn results which is a primary tool used to discriminate material fire performance by governing bodies such as UL, ASTM, FAA and the like. As such these materials do not meet such performance standards

10
15
20

Table 5 Torque Rheology

Extrusion Torque Rheology					
Formulation	Nylon12	1	2	3	4
Torque (Nm)	105	100	95	91	87
Batch mixer torque rheology					
Formulation	Nylon12	3	5	6	
Torque (Nm)	47	44	47	49	

- 41 -

Table 6 Mechanical Performance

Formulation	Nylon12	Nylon12 FR	1	2	3	4
Modulus (MPa)	1110	1712	1187	1227	1470	1700
Tensile Strength (MPa)	36	48	53	52.3	57	44.6
Impact (k/m ²)	4006	2200	6200	8100	6700	3700

Table 7 Fire Testing Cone Results

5

Formulation	Peak Heat Rel ^d kW/m ²	Mass Loss Rate g/m ² s	CO Prod ⁿ Kg/Kg	CO ₂ Prod ⁿ Kg/Kg	SEA (Smoke) m ² /Kg
Nylon 12 FR	1800	18.6	0.01	1.2	100
Nylon12	1344	17.1	0.03	1.6	385
1	740	13.3	0.01	1.0	360
2	620	12.8	0.02	1.5	382
3	536	10.8	0.02	1.5	382
4	447	10.0	0.02	1.5	410

Table 8 Vertical Burn Results

Formulation	UL94 (3.2 mm)	FAA (1.6 mm)
Nylon 12 FR	VO	Pass
Nylon12 LV	HB	Fail
1	V2	Fail
2	V2	Fail
3	V1	Fail
4	V1	Fail

- 42 -

5

Example 8:

Processing (Table 9), XRD (Figure 3),
mechanical (Table 10) and fire performance
(Table 11-14) of nylon12 modified with
commercially available clay and flame
retarding additives (melamine cyanurate)
during melt processing

10

The following example indicates that the processing rheology of Nylon 12 is not effected by the melt dispersion of commercially available 'organoclay' at least partially on a nanometer scale (XRD) and flame retardant. This dispersion results in improved mechanical performance reduced heat release results via cone calorimetry and vertical burn performance for specimens greater than 1.6 mm thickness compared with conventionally flame retarded nylon12. Although samples of 0.75mm thickness provide good smoke and toxic gas release results they fail FAA type 12 sec vertical burn testing and perform badly in radiant panel tests. This indicates that the strategy is not satisfactory to meet the performance of thin parts to the performance requirements of governing bodies such as the FAA.

15

20

Table 9 Processing Rheology

Formulation	Torque (Nm)
Nylon 12	105
7	102
8	104
9	107

- 43 -

Table 10 Mechanical Performance

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Elongation at break (%)	Notched Impact Strength (J/m ²)
Nylon12	1110	36	640	4600
Nylon12 FR	1712	48.1	77	2100
7	1505	38.5	54	3100
8	1471	38.1	222	4100
9	1380	38.1	291	4600

Standard Deviation -Modulus<4%, Strength<3%,
Elongation<10%, Impact <11%

5

Table 11 Fire Testing Cone Calorimetry

Formulation	Peak Heat Rel ^d kW/m ²	Mass Loss Rate g/m ² s	CO Prod ⁿ Kg/Kg	CO ₂ Prod ⁿ Kg/Kg	SEA (Smoke) m ² /Kg
Nylon 12 FR	1800	18.6	0.01	1.2	100
Nylon12	1344	17.1	0.03	1.6	385
7	670	13.9	0.01	1.6	220
8	695	14.1	0.01	1.6	240
9	782	16.1	0.01	1.7	280

Table 12 Vertical Burn Results

10

Formulation	UL94 (3.2 mm)	FAA 12s (1.6 mm)	FAA 12s (0.75 mm)
Nylon 12 FR	V0	Pass	Fail
Nylon12	HB	Fail	Fail
7	V0	Pass	Fail
8	V0	Pass	Fail
9	V0	Pass	Fail

- 44 -

Table 13 Vertical Burn, Radiant Panel and Smoke Test
Results (0.75mm)

Formulation	FAA 12s (0.75mm)	Smoke Ds	Radiant Panel
9	Fail	4.88	Full length burn
8	Fail	11.86	Full length burn
7	Fail	21.45	Full length burn

5

Table 14 Toxic Gas Emission

Toxic Gas (ppm)	Formulation		
	9	8	7
HF	3	3	5
HCl	1	1	3
HCN	4	4	4
H ₂ S	-	-	-
NO _x	2	2	1
HBr	1	1	1
PO ₄	-	-	-
SO ₂	1	1	1

- 45 -

Example 9: Processing rheology (Table 15), XRD (Figure 4), mechanical (Table 16) and fire performance (Table 17-19) of nylon12 modified with IOH2 incorporating montmorillonite modified with melamine hydrochloride/melamine and flame retarding additives (melamine cyanurate) during melt processing

The following example indicates that the processing rheology of Nylon 12 is not effected by the melt dispersion of IOH2 and flame retardant at least partially on a nanometre scale (XRD). Such dispersion results in improved mechanical and vertical burn results compared with conventionally flame retarded nylon12. Samples of 0.75 mm provide good smoke and toxic gas release results, pass FAA type 12s vertical burn tests and perform better in radiant panel tests. It is known to those in the art that flame retarding thin polymeric based materials is much more difficult than flame retarding thicker materials and as such meeting performance requirements at thin thickness is an indication of superior fire retarding performance.

25 Table 15 Processing Rheology

Formulation	Extruder Torque (Nm)
Nylon 12	105
11	105
12	106
13	103

- 46 -

Table 16 Mechanical Performance

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Elongation at break (%)	Notched Impact Strength (J/m ²)
Nylon12	1110	36	640	4600
Nylon12 FR	1712	48.1	77	2100
11	1443	39.7	140	3900
12	1398	39.0	215	4200
13	1349	38.9	375	4700

Standard Deviation -Modulus<3%, Strength<3%,
Elongation<8%, Impact <9%

5

Table 17 Fire Performance - Vertical Burn

Formulation	UL94 (3.2 mm)	12s FAA (1.6 mm)	12s FAA (0.75 mm)	60s FAA (0.75mm)
Nylon12 FR	VO	Pass	Fail	Fail
Nylon12	HB	Fail	Fail	Fail
11	VO	Pass	Pass	Pass
12	VO	Pass	Pass	Pass
13	VO	Pass	Pass	Pass

- 47 -

Table 18 Fire Performance (0.75mm)

Formulation	FAA 12s Vertical Burn Extinguishment time Burn length Drip Extinguishment time	Smoke Ds	Radiant Panel Extinguishment time & Burn length
11	4.9 s 46mm 0 s	6.79	5 sec 25 mm
12	2 s 19mm 0 s	9.83	3 sec 25mm
13	0 s 21 mm 0 s	3.31	1 sec 12.5 mm

Table 19 Toxic Gas Emission

5

Toxic Gas Emission (ppm)	Formulation		
	13	12	11
HF	6	4	3
HCl	1	1	1
HCN	8	7	7
H ₂ S	-	-	-
NO _x	3	2	2
HBr	1	1	1
PO ₄	-	-	-
SO ₂	1	1	1

5

Example 10: The following example illustrates the effect of different processing parameters on the mechanical performance (Table 20) and vertical burn performance (Table 21) of formulation 13 which incorporates IOH2 + conventional flame retardant melamine cyanurate

10 Results indicate the robustness of the formulation in terms of mechanical and fire performance to different processing conditions such as through-put, temperature profile, screw speed for the given screw and barrel configuration provided in Figure 1.

15

Table 20 Mechanical Performance

Conditions			Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)
Processing Temp. (°C)	Screw speed (rpm)	Through-put (Kg/h)			
180	300	1.5	1300	37.6	5100
190	300	1.5	1420	37.9	5300
200	300	1.5	1420	38.4	4800
210	300	1.5	1520	38.8	4600
200	150	1.5	1500	37.7	5300
200	400	1.5	1530	39.6	4100
200	300	15	1540	39.4	4100

Standard Deviation -Modulus<3%, Strength<3%, Impact <9%

- 49 -

**Table 21 FAA 12s Vertical Burn Performance
(0.75mm thickness)**

Conditions			Result	Flame out Time (sec)
Processing Temp.(°C)	Screw speed (rpm)	Through-put (Kg/h)		
180	300	1.5	Pass	5
190	300	1.5	Pass	4
200	300	1.5	Pass	2
210	300	1.5	Pass	6
200	150	1.5	Pass	2
200	400	1.5	Pass	7
200	300	15	Pass	3

5 Example 11: The following example illustrates the effect of different IOH2 (Example 2) and melamine cyanurate concentrations on mechanical and vertical burn performance of nylon12 (Table 22)

10

Results indicate that preferably more than 10% melamine cyanurate is required to pass FAA 12 s vertical burn test requirements at 0.75 mm thickness. Results also indicate that unlike classically flame retarded nylon12 this fire 15 performance is achievable whilst maintaining excellent mechanical properties relative to nylon12.

- 50 -

Table 22 Performance of Formulations incorporating different concentrations of IOH2 and Melamine cyanurate

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)	FAA 12s Vertical burn (0.75mm) Ext. Time (s)
Nylon12	1100	36	4600	Fail (62)
Nylon12 FR	1712	48.1	2100	Fail (24)
11	1443	39.7	3900	Pass (5)
12	1398	39.0	4200	Pass (5)
13	1349	38.9	4700	Pass (2)
14	1480	37.9	4200	Pass (14)
15	1410	39.4	4400	Pass (7)
16	1386	40.1	4800	Pass (6)
17	1483	37.9	3900	Fail (18)
18	1476	39.4	5050	Fail (19)
19	1404	40.1	5200	Fail (19)
20	1445	37.8	4200	Fail (32)
21	1420	39.7	4500	Fail (28)
22	1361	40.1	5200	Fail (32)

5 Example 12: The following example illustrates the effect of different conventional flame retardants on the performance (Table 23) of nylon12 incorporating an IOH2 (Example 2)

10 The results presented in Table 23 demonstrate that materials incorporating the IOH and melamine cyanurate provide both excellent mechanical and fire performance. Formulations containing melamine phthalate and pentaerythritol phosphate also provide excellent fire performance with lower mechanical performance. Samples containing IOH with melamine cyanurate and Mg(OH)₂ provide the excellent mechanical performance in terms of impact, modulus, and strength also excellent vertical burn performance.

15

Table 23 Performance of formulations incorporation IOH2 and various conventional flame retardants

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)	FAA 12 s vertical burn (0.75mm) Ext. Time (sec)	UL 94 3.2 mm
12	1460	39	4800	Pass (2)	V0
23	1500	41	3900	Fail (31)	V2
24	1540	41.9	2500	Fail (26)	V2
25	1500	40.4	3000	Fail (29)	V2
26	-	-	-	Pass (7)	V0
27	1410	41.0	4100	Fail (24)	V2
28	1420	43.5	1500	Fail (32)	V2
29	1160	43.6	800	Pass (10)	V0
30	1628	43.6	4800	Pass (4)	V0

5

Example 13: The following example illustrates the effect of removing components of the fire resistant formulation on resultant fire performance (Table 24)

10

The results indicate that removal of either the modified inorganic-organic hybrid or melamine cyanurate from the formulation provides unsatisfactory vertical burn performance following FAA 12 s type testing at 0.75mm thickness.

15

Table 24 FAA type Vertical Burn Performance (0.75mm)

Formulation	Ext. Time (s)	FAA requirement
Nylon12	65 ± 9	Fail
31	31 ± 4	Fail
32	32 ± 13	Fail
15	7 ± 4	Pass

Example 14: The following example illustrates the mechanical and 12s vertical burn performance (Table 25) and cone calorimetry results (Table 26) of Nylon12 formulations prepared with IOH2 (Example 2), melamine cyanurate and magnesium hydroxide. Table 27 provides radiant panel, smoke, and 60s FAA type vertical burn results for the above mentioned formulations. Mechanical and vertical burn performance of Nylon12 formulations incorporating IOH2, melamine cyanurate and magnesium hydroxide of different surface functionality and particle size distribution is provided in Table 28.

Results from Example 14 show that excellent processability, mechanical, vertical burn, and heat release results are obtainable with formulations incorporating IOH2, melamine cyanurate and low concentrations of magnesium hydroxide in particular formulations incorporating IOH dispersed at least partially on a nanometre scale, melamine cyanurate and 2.5% magnesium hydroxide which provides excellent mechanical, vertical burn and peak and average heat release results. The results also indicate that Mg(OH₂) of different grades may be employed in conjunction with IOH2 and melamine cyanurate to produce formulations with excellent processability, mechanical and fire performance.

Table 25 Mechanical Performance of nylon materials with various amounts of IOH₂ and conventional flame retardants

Formulation	MFI (g/min)	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)	FAA 12s Vertical burn Ext. Time (s) (0.75mm)
Nylon12	44	1100	36	4600	Fail (62)
Nylon12 FR	32	1712	48.1	2100	Fail (24)
33	12.6	1470	41.8	4500	Fail (18)
34	12.0	1460	41.1	4700	Pass (10)
35	11.5	1430	39.9	5200	Pass (9)
36	13.4	1578	43	3800	Pass (6)
30	13.5	1509	42	4800	Pass (4)
37	13.5	1543	40.5	5300	Pass (6)
38	13.4	1529	41	3900	Fail (41)
39	13	1520	40.6	4200	Fail (19)
40	13.1	1510	41.6	4600	Pass (4)

5

Table 26 Cone Calorimeter Heat Release Results

Formulation	Peak Heat Release (kW/m ²)	300s Average Heat Release (kW/m ²)
Nylon12	1100	748
Nylon12 FR	1712	640
18	1314	707
21	1643	680
12	1595	676
39	1147	552
30	1001	578
34	885	491

- 54 -

Table 27 Comparison of fire performance of various formulations containing IOH₂ dispersed at least partially on a nanometre scale, melamine cyanurate and optionally magnesium hydroxide H7

5

Formulation	Radiant Panel Extinguishment time & Burn length (average)	Smoke Ds	Toxic Gas (FAA requirement)	FAA 60 Second Vertical burn (0.75mm) (Extinguishment time seconds)
Nylon12	-	21	Pass	-
22	-	11.7	Pass	-
21	-	10.4	Pass	-
20	-	7.8	Pass	-
19	-	11.3	Pass	-
18	-	11.4	Pass	Fail (20)
17	-	8.1	Pass	Pass (9)
13	1 second 12.4 mm	14.5	Pass	Pass (0)
12	-	14.4	Pass	Pass (0)
11	-	7.5	Pass	Fail (133)
39	-	15	Pass	Fail (58)
30	-	14.5	Pass	Pass (15)
34	2.5 second 15.0 mm	11.3	Pass	Pass (7)

- 55 -

**Table 28 Performance of materials incorporating IOH2
melamine cyanurate and Mg(OH)2 with various particle size
and surface functionality**

Formulation	MFI (g/min)	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)	FAA 12s Vertical burn Ext. Time (s) (0.75mm)
34	13.5	1480	40.4	5100	Pass (6)
41	11.5	1420	41	5000	Pass (6)
42	16.2	1470	40.2	5300	Pass (13)
44	12.4	1470	40.4	5300	Pass (14)

5

Example 15: The following example illustrates the mechanical and vertical burn performance

(Table 29) of Nylon12 formulations prepared

with the inorganic-organic hybrids outlined

in Examples 1, 2 & 4 and melamine cyanurate

10 The results indicate superior fire performance of nylon12 formulations containing the intercalated and modified IOH (Examples 2 and 4) compared with that prepared with just 15 melamine hydrochloride modified IOH (Example 1).

Table 29 Mechanical and Vertical Burn Performance

Formulation	Tensile Strength (MPa)	Tensile Modulus (MPa)	Notched Impact Strength J/m ²	0.75 mm FAA 12 sec Vertical Burn (Ext. time sec)
44	41.7	1490	5000	Fail (22)
45	39.5	1531	4100	Pass (12)
46	40.1	1580	4600	Pass (2)
47	39.2	1550	4100	Pass (5)
18	40.4	1590	4700	Fail (19)
12	39.3	1628	4000	Pass (3)

Standard Deviation -Modulus<5%, Strength<5%, Impact <10%

Example 16: The following example illustrates the performance of nylon6 and nylon66 formulations incorporating IOH2 and melamine cyanurate

5

The results indicate that IOH2 at least partially dispersed on a nanometre scale in conjunction with melamine cyanurate provides excellent mechanical and 10 vertical burn performance relative to nylon6 and nylon66.

Table 30. Mechanical and Vertical Burn Performance

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)	FAA 12s Vertical burn Ext. Time (s) (0.75mm)
Nylon6	2720	76	1900	Fail (61)
48	2970	73.5	2000	Pass (1)
Nylon66	2890	83.5	1900	Fail (65)
49	3500	67	1900	Pass (1)

15 Example 17: The following example shows the XRD of

nylon 12 formulations incorporating modified and intercalated hectorite (Example 6) dispersed at least partially on 20 a nanometre scale (Figure 5) and melamine cyanurate and the formulations vertical burn performance (Table 31)

The XRD results indicate that hectorite is modified owing to its larger intergallery spacing compared with the 25 starting material. Nylon12 incorporating IOH5 at least partially dispersed on a nanometre scale (Figure 5) and melamine cyanurate show excellent fire performance.

Table 31 Vertical Burn Performance

Formulation	FAA 12s Vertical burn, Ext. Time (s)(0.75mm)
Nylon12	Fail (68)
50	Pass (2)

Example 18: This example shows the rheology (Table 32) and mechanical and vertical burn performance (Table 33) of formulations incorporating IOH2, conventional flame retardant and minor processing additives.

5 This example illustrates that reductions in viscosity across a range of shear rates of the formulations incorporating nylon12, IOH2 and conventional flame retardants through the addition of (additional) minor processing additives during processing. This reduction in 10 viscosity is possible without a significant reduction in mechanical performance and generally without compromising fire performance particularly under the stringent 15 conditions required to fire retard thin materials to meet performance standards outlined by various regulatory 20 bodies.

- 58 -

Table 32 Rheology of formulations at different shear rates and corresponding MFI data

Formulation	Shear rate				MFI
	10^{-2}	10^{-1}	10^0	10^1	
	Viscosity (Pas)				g/min
Nylon12	223	169	106	108	35
13	13100	1750	300	124	29
34	719	624	560	502	13
51	4800	1040	226	128	34
52	1920	6590	1560	95	39
53	1100	865	168	95	39
54	554	865	162	95	41
55	98300	1930	335	143	33
56	13500	1870	284	106	31

5 **Table 33 Mechanical and Vertical Burn Performance**

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength (J/m ²)	0.75 mm FAA 12 sec Vertical Burn (Extinguishment time (s))
Nylon12	1100	36	4600	Fail (62)
13	1349	38.9	4700	Pass (2)
34	1480	40.4	5100	Pass (6)
51	1215	35.8	3500	Pass (3)
52	1165	35.5	3500	Pass (2)
53	1233	36.4	3500	Pass (13)
54	1176	35.3	3300	Fail (25)
55	1168	33.3	3300	Pass (8)
56	1241	35	3700	Pass (10)

- 59 -

Example 19: This example provides the mechanical and fire performance (Table 34) of nylon12 formulations incorporating IOH2, conventional flame retardants and minor component of stabilizer.

5

The results indicate that the mechanical and vertical burn performance of formulations containing nylon12, IOH2 conventional flame retardant is not significantly reduced 10 by addition of additional stabilizer to the formulation during compounding.

Table 34 Mechanical and Vertical Burn Performance

Formulation	Tensile Modulus (MPa)	Tensile Strength (MPa)	Notched Impact Strength J/m ²	0.75 mm FAA 12 sec Vertical Burn (Extinguishment time (s))
Nylon12	1100	36	4600	Fail (62)
13	1349	38.9	4700	Pass (2)
57	1394	39.1	4800	Pass (4)

15

Example 20: This example shows that formulations incorporating IOH's may not only be fabricated into materials, components and parts of components by processes such as extrusion, injection moulding, compression moulding and alike but also by low shear processes such as rotational moulding (Figure 6) and selective laser sintering.

20

25 Figure 6 provides examples of components manufactured by rotational moulding employing formulations incorporating IOH2, melamine cyanurate optionally magnesium hydroxide and other additives such as but not limited to formulation 13 and 34. The examples illustrate that such formulations 30 show suitable thermal / oxidative stability and melt

- 60 -

rheology for manufacturing components under low shear and thermally demanding environments.

It will be appreciated by persons skilled in the
5 art that numerous variations and/or modifications may be
made to the invention as shown in the specific embodiments
without departing from the spirit or scope of the
invention as broadly described. The present embodiments
are, therefore, to be considered in all respects as
10 illustrative and not restrictive.

- 61 -

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An inorganic-organic hybrid (IOH) which comprises:
 - 5 (i) an expandable or swellable layered inorganic component; and
 - (ii) an organic component including at least one ionic organic component and one or more neutral organic components which are intercalated between and/or
- 10 15 associated with the layer(s) of the inorganic component, the ionic or neutral organic components being capable of decomposing or subliming endothermically, and/or releasing volatiles with low combustibility on decomposition and/or inducing charring of organic species during thermal decomposition or combustion.
2. An IOH according to claim 1, in which the inorganic component is rendered positively or negatively charged due to isomorphic substitution of elements within
- 20 25 the layers.
3. An IOH according to claim 1 or claim 2, in which the inorganic component is selected from a 1:1 layered silicate structure, a 2:1 layered silicate structure, a double hydroxide of the general formula $Mg_6Al_{3.4}(OH)_{18.8}(CO_3)_{1.7} \cdot H_2O$ and a synthetically prepared layered material.
4. An IOH according to any one of claims 1 to 3, in
- 30 35 which the inorganic compound is a naturally occurring or a synthetic analogue of a phyllosilicate.
5. An IOH according to claim 4, in which the naturally occurring or synthetic analogue of a
- 35 phyllosilicate is a smectite clay.

- 62 -

6. An IOH according to claim 5, in which the smectite clay is selected from montmorillonite, nontronite, beidellite, volkonskoite, hectorite, bentonite, saponite, saucinite, magadiite, kanyaite, laponite, vermiculite, synthetic micromica and synthetic hectorite.

5

7. An IOH according to claim 5 or claim 8, in which the naturally occurring phyllosilicate is selected from bentonite, montmorillonite and hectorite.

10

8. An IOH according to any one of claims 4 to 7, in which the phyllosilicate has a platelet thickness less than about 5 nanometers and an aspect ratio greater than 15 about 10:1.

9. An IOH according to claim 8, in which the aspect ratio is greater than about 50:1.

20

10. An IOH according to claim 8 or claim 9, in which the aspect ratio is greater than about 100:1.

11. An IOH according to any one of claims 1 to 10, in which the inorganic component includes interlayer or 25 exchangeable metal cations to balance the charge.

30

12. An IOH according to claim 11, in which the metal cation is selected from an alkali metal and alkali earth metal.

13. An IOH according to claim 12, in which the alkali or alkali earth metal is selected from Na^+ , K^+ , Mg^{2+} and Ca^{2+} .

35

14. An IOH according to any one of claims 11 to 13, in which the cation exchange capacity of the inorganic

- 63 -

component is less than about 400 milliequivalents per 100 grams.

15. An IOH according to any one of claims 11 to 14,
5 in which the ionic organic component is exchanged with the
exchangeable metal ions of the inorganic component.

16. An IOH according to any one of claims 1 to 15,
10 in which the ionic species contains onium ion(s).

17. An IOH according to claim 16, in which the ionic
15 species containing onium ion(s) is an ammonium,
phosphonium or sulfonium derivative of an aliphatic,
aromatic or aryl-aliphatic amine, phosphine or sulfide.

18. An IOH according to any one of claims 1 to 17,
20 in which the ionic or neutral organic component is a
neutral or ionic derivative of a nitrogen based molecule.

19. An IOH according to claim 18, in which the
25 nitrogen based molecule is a triazine based species.

20. An IOH according to claim 19, in which the
triazine based species is selected from melamine,
25 triphenyl melamine, melam (1,3,5-triazine-2,4,6-triamine-
n-(4,6-diamino-1,3,5-triazine-yl)), melem ((-2,5,8-
triamino-1,3,4,6,7,9,9b-heptaazaphenalenene)), melon
30 (poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalenene-2,5-
diyl)imino}), bis and triaziridinyltriazine,
trimethylsilyltriazine, melamine cyanurate, melamine
phthalate, melamine phosphate, melamine phosphite,
melamine phthalimide, dimelamine phosphate, phosphazines,
35 low molecular weight polymers with triazine and
phosphazine repeat units and isocyanuric acid and salts or
derivatives thereof.

- 64 -

21. An IOH according to claim 20, in which isocyanuric acid and salts or derivatives thereof are selected from isocyanuric acid, cyanuric acid, triethyl cyanurate, melamine cyanurate, triglycidylcyanurate, triallyl isocyanurate, trichloroisocyanuric acid, 1,3,5-tris(2-hydroxyethyl)triazine-2,4,6-trione, hexamethylenetetramine.melam cyanurate, melem cyanurate and melon cyanurate.
- 10 22. An IOH according to any one of claims 18 to 21, in which the organic component is a derivative of phosphoric acid or boric acid.
- 15 23. An IOH according to claim 22; in which the derivative of phosphoric acid or boric acid is selected from ammonia polyphosphate, melamine polyphosphate and melamine phosphate ammonium borate.
- 20 24. An IOH according to any one of claims 1 to 23, in which the ionic organic component is used in combination with other ionic compounds which are capable of improving compatibility and dispersion between the inorganic and organic components.
- 25 25. An IOH according to claim 24, in which the other ionic compound is an amphiphilic molecule that incorporates a hydrophilic ionic group along with hydrophobic alkyl or aromatic moieties.
- 30 26. An IOH according to any one of the preceding claims, which further comprises one or more coupling reagents.
- 35 27. An IOH according to claim 26, in which the coupling reagent is selected from an organically functionalised silane, zirconate and titanate.

28. An IOH according to claim 27, in which the silane coupling reagent is tri-alkoxy, acetoxy or halosilanes functionalised with amino, epoxy, isocyanate, hydroxyl, thiol, mercapto and/or methacryl reactive moieties or modified to incorporate functional groups based on triazine derivatives, long chain alkyl, aromatic or alkylaromatic moieties.

5

29. A method for the preparation of the IOH defined in any one of claims 1 to 28, which comprises mixing components (i) and (ii) defined in any one of claims 1 to 28 or constituents thereof in one or more steps.

10

30. A method according to claim 29, in which mixing is achieved using melt, solution or powder processing.

15

31. A method according to claim 29 or claim 30, in which the mixing is achieved using solution processing.

20

32. Use of the IOH defined in any one of claims 1 to 28 as a fire resistant material.

33. A fire resistant formulation which comprises:

25 (i) the IOH defined in any one of claims 1 to 28; and

(ii) one or more flame retardants.

34. A formulation according to claim 33, in which the flame retardant is selected from phosphorus derivatives, nitrogen containing derivatives, molecules containing borate functional groups, molecules containing two or more alcohol groups, molecules which endothermically release non-combustible decomposition gases and expandable graphite.

35

35. A formulation according to claim 34, in which the phosphorus derivatives are selected from melamine

phosphate, dimelamine phosphate, melamine polyphosphate, ammonia phosphate, ammonia polyphosphate, pentaerythritol phosphate, melamine phosphite and triphenyl phosphine.

5 36. A formulation according to claim 34 or claim 35, in which the nitrogen containing derivatives are selected from melamine, melamine cyanurate, melamine phthalate, melamine phthalimide, melam, melem, melon, melam cyanurate, melem cyanurate, melon cyanurate, hexamethylene 10 tetraamine, imidazole, adenine, guanine, cytosine and thymine.

15 37. A formulation according to any one of claims 34 to 36, in which the molecules containing borate functional groups are selected from ammonia borate and zinc borate.

20 38. A formulation according to any one of claims 34 to 37, in which the molecules containing two or more alcohol groups are selected from pentaerythritol, polyethylene alcohol, polyglycols and carbohydrates.

25 39. A formulation according to any one of claims 34 to 38, in which the molecules which endothermically release non-combustible decomposition gases are selected from magnesium hydroxide and aluminum hydroxide.

30 40. A method for the preparation of the fire resistant formulation defined in any one of claims 33 to 39, which comprises mixing components (i) and (ii) as defined in any one of claims 1 to 28 or constituents thereof in one or more steps.

35 41. A method according to claim 40, in which mixing is achieved using melt, solution or powder processing.

42. A method according to claim 40 or claim 41, in which the mixing is achieved using melt processing in a

twin screw extruder or batch mixer; or powder processing using a high shear powder mixer or milling procedures.

43. A polyamide fire resistant formulation which
5 comprises either:

(A) (i) the IOH defined in any one of claims 1 to 28; and

(ii) a polyamide based matrix; or

(B) (i) the fire resistant formulation defined
10 in any one of claims 33 to 39; and

(ii) a polyamide based matrix.

44. A formulation according to claim 43, in which
the polyamide based matrix comprises generic groups with
15 repeat units based on amides selected from Nylon4, Nylon6,
Nylon7, Nylon 11, Nylon12, Nylon46, Nylon66, Nylon 68,
Nylon610, Nylon612 and aromatic polyamides and co-
polymers, blends or alloys thereof.

20 45. A formulation according to claim 43 or claim 44,
in which the polyamide based matrix is selected from
Nylon12, Nylon6 and Nylon66 and co-polymers, alloys or
blends thereof.

25 46. A formulation according to any one of claims 43 to 45, which further comprises one or more additives.

47. A formulation according to claim 46, in which
the additives are selected from polymeric stabilisers;
30 lubricants; antioxidants; pigments, dyes or other
additives to alter the materials optical properties or
colour; conductive fillers or fibers; release agents; slip
agents; plasticisers; antibacterial or fungal agents; and
processing agents.

- 68 -

48. A formulation according to claim 47, in which the polymeric stabiliser is a UV, light or thermal stabilizer.

5 49. A formulation according to claim 47 or claim 48, in which the processing agents are selected from dispersing reagents, foaming or blowing agents, surfactants, waxes, coupling reagents, rheology modifiers, film forming reagents and free radical generating 10 reagents.

50. A formulation according to any one of claims 43 to 49, in which the polyamide based matrix is Nylon12, Nylon6 and/or Nylon66; the IOH is montmorillonite or 15 hectorite modified with melamine hydrochloride and/or melamine cyanurate hydrochloride and/or melamine and/or melamine cyanurate; and the flame retardant is melamine cyanurate and/or magnesium hydroxide; and the additive is a processing agent and/or a polymeric stabiliser.

20 51. A formulation according to any one of claims 46 to 50, in which the polyamide based matrix is present in an amount of about 45 to about 95% w/w, the IOH is present in an amount less than about 25% w/w and the flame 25 retardant and/or additives are present in an amount less than about 30% w/w.

52. A formulation according to any one of claims 46 to 51, in which the polyamide based matrix is present in 30 an amount greater than about 75% w/w, the IOH is present in an amount less than about 3% w/w, the melamine cyanurate flame retardant is present in an amount of about 11 to about 15% w/w and additives are present in an amount of about less than about 4% w/w.

35 53. A formulation according to any one of claims 46 to 51, in which the polyamide based matrix is present in

- 69 -

an amount greater than about 75% w/w, the IOH is present in an amount less than about 3% w/w, the melamine cyanurate flame retardant is present in an amount of about 11 and about 15% w/w, magnesium hydroxide flame retardant present in an amount of about 1 and about 5% w/w and additives are present in an amount less than about 4% w/w.

54. A method for the preparation of the polyamide
fire resistant formulation defined in any one of claims 43
10 to 53, which comprises dispersing the IOH as defined in
any one of claims 1 to 28 or the fire resistant
formulation defined in any one of claims 33 to 39 or
constituents thereof into the polyamide based matrix in
one or more steps.

15

55. A method according to claim 54, in which at least some of the components are ground prior to mixing.

56. A method according to claim 55, in which the components are ground to a particle size less than about 200 microns.

57. A method according to claim 55 or claim 56, in which dispersion is achieved using melt, solution or powder processing

58. A method according to any one of claims 55 to 57, in which the dispersion is achieved using melt processing in a single or twin screw extruder, batch mixer or continuous compounder.

59. A method according to claim 58, in which the melt processing is conducted in a twin screw extruder.

35

60. A method according to any one of claims 54 to 59, in which the dispersion occurs at a sufficient shear

- 70 -

rate, shear stress and residence time to disperse the IOH at least partially on a nanometer scale.

61. A fire resistant article or parts thereof which
5 is composed wholly or partly of the IOH as defined in any one of claims 1 to 28 and/or fire resistant formulation defined in any one of claims 33 to 39 and claims 43 to 53.

62. A fire resistant article or parts thereof as
10 defined in claim 61, which is used in transport, building, construction, electrical or optical applications.

63. A fire resistant article or parts thereof as
defined in claim 62, in which the transport application is
15 air, automotive, aerospace or nautical.

64. A fire resistant article or parts thereof as
defined in any one of claims 61 to 63, which is a hollow article or sheet.

20
65. A fire resistant article or parts thereof as
defined in any one of claims 61 to 64 which is selected from pipes, ducts, fabric, carpet, cables, wires, fibres, Environmental control systems, stowage bin hinge covers, 25 cable trays, ECS duct spuds, latches, brackets, passenger surface units and thermoplastic laminate sheet.

66. A fire resistant hollow article or parts thereof which is composed wholly or partly of the fire resistant 30 formulation defined in claim 52 or claim 53 and manufactured by rotational moulding or extrusion.

67. A fire resistant fibre, fabric, carpet or parts thereof which is composed wholly or partly of the fire 35 resistant formulation defined in claim 52 or claim 53 and manufactured by melt spinning or extrusion.

68. A fire resistant article or parts thereof which is composed wholly or partly of the formulation defined in claim 52 or claim 53 and manufactured by sintering.

5 69. A fire resistant article or parts thereof which is composed wholly or partly of the fire resistant formulation defined in claim 52 or claim 53 and manufactured by injection or compression moulding.

10 70. A method of preparing the fire resistant article or parts thereof defined in any one of claims 57 to 69, which comprises moulding or forming the IOH as defined in any one of claims 1 to 28 and/or the fire resistant formulation or constituents thereof as defined in any one 15 of claims 33 to 39 and claims 43 to 49.

71. A method according to claim 70, in which the moulding or forming is carried out using extrusion, injection moulding, compression moulding, rotational 20 moulding, blow moulding, sintering, thermoforming, calendering or combinations thereof.

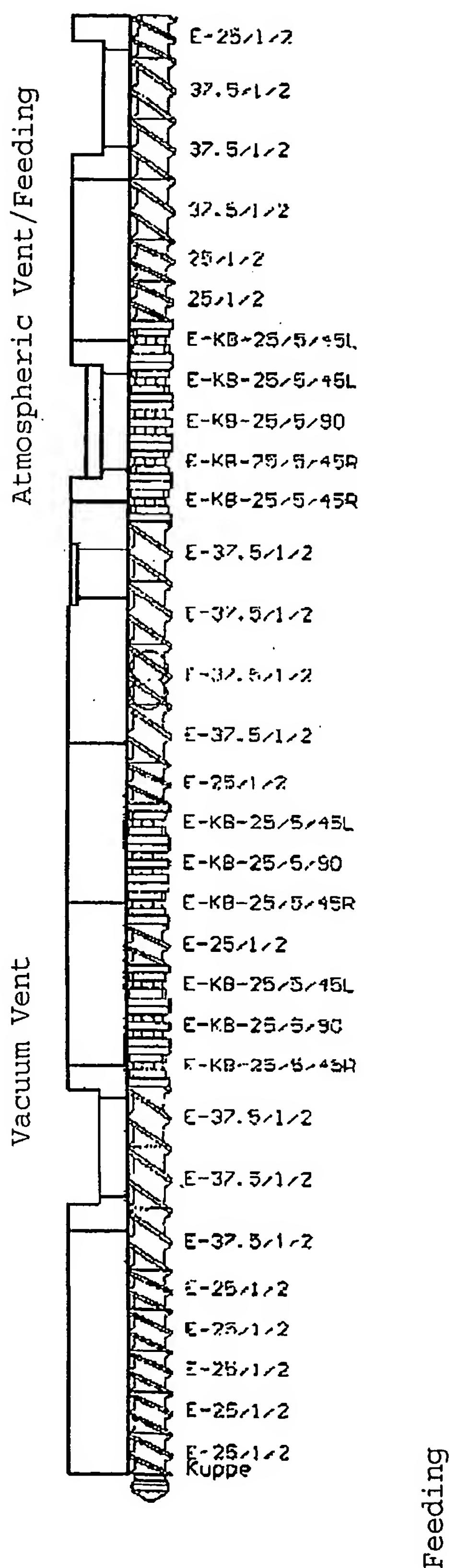


Figure 1

2 / 6

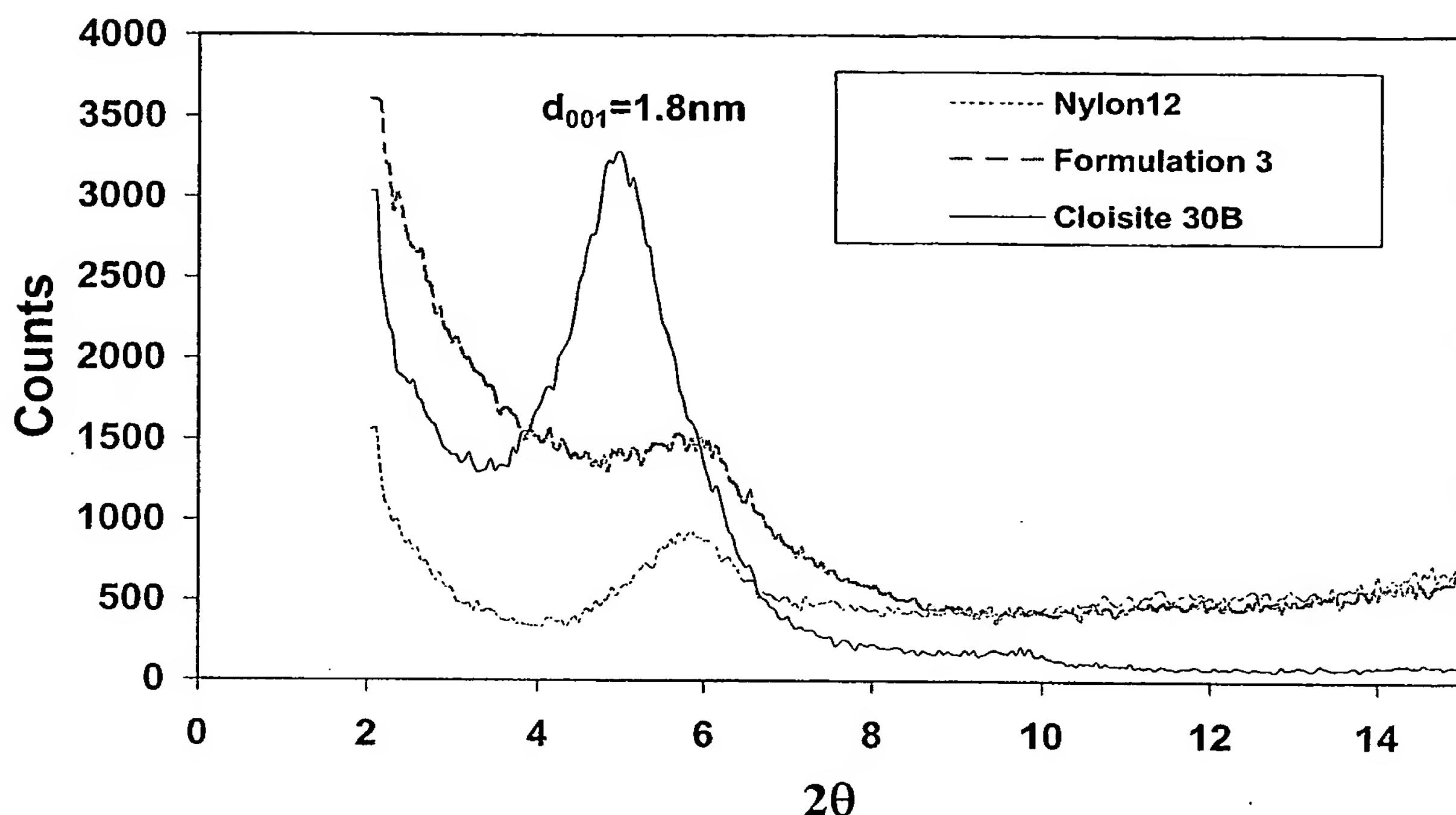


Figure 2

3/6

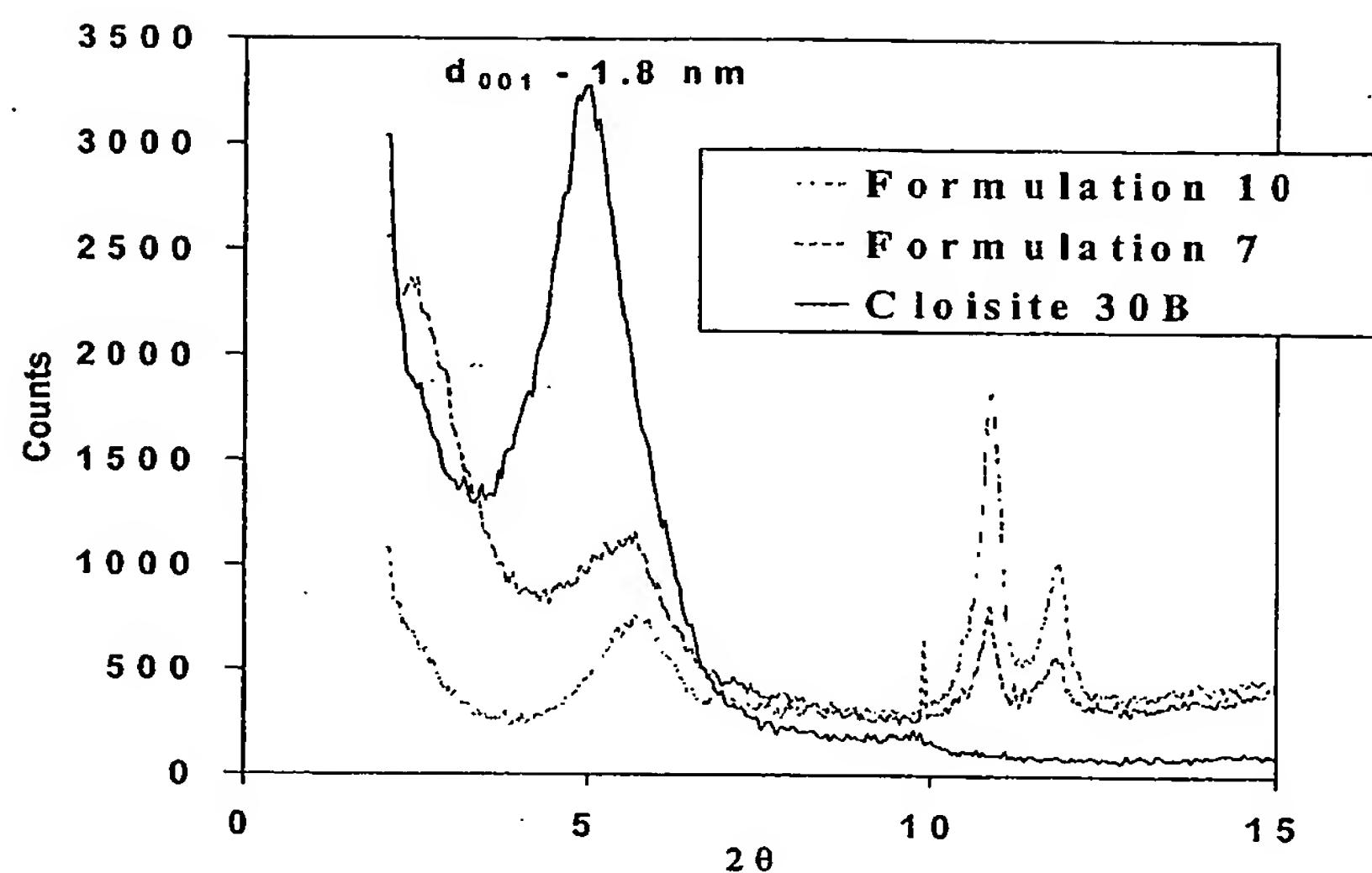


Figure 3

4/6

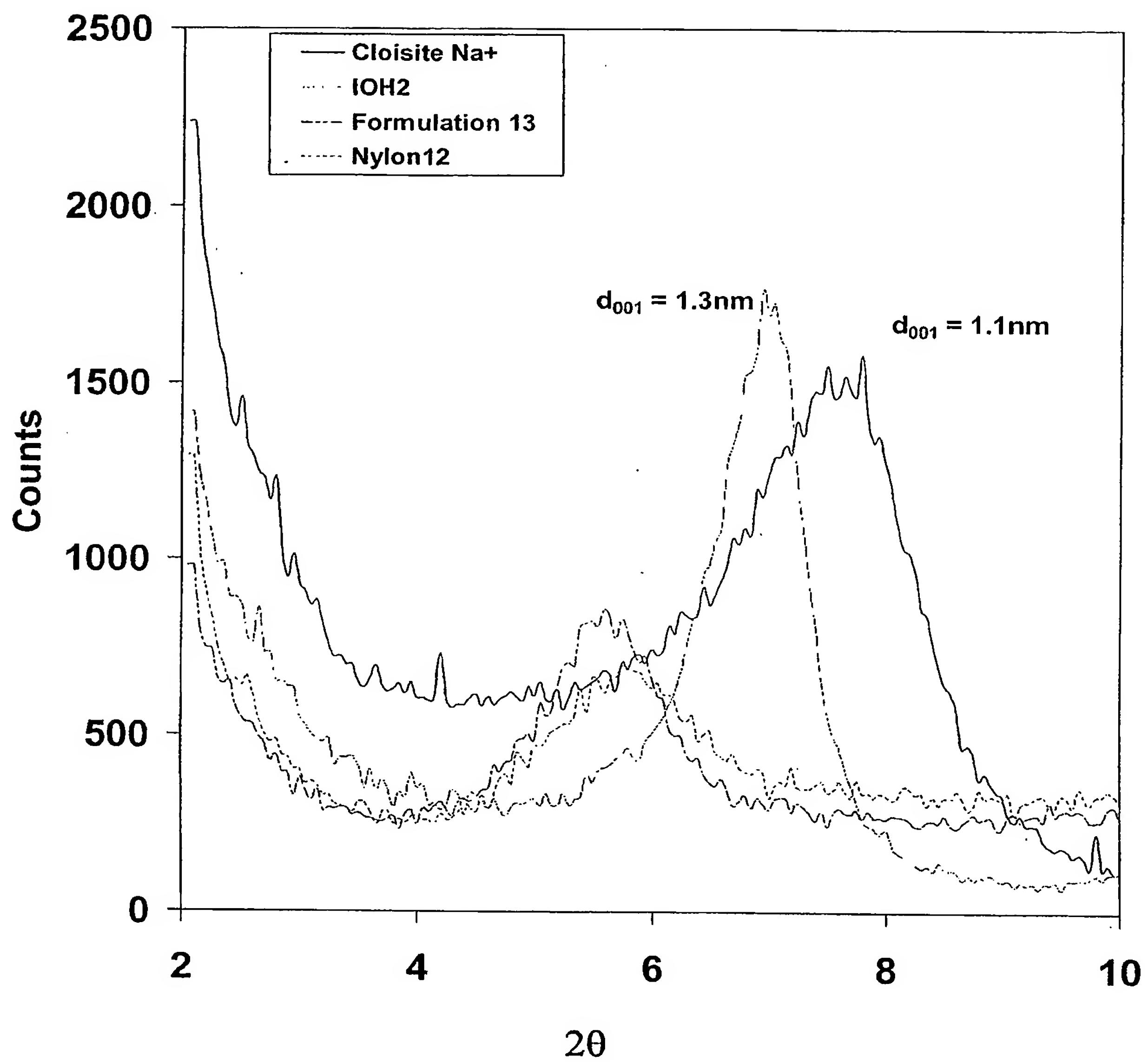


Figure 4

5/6

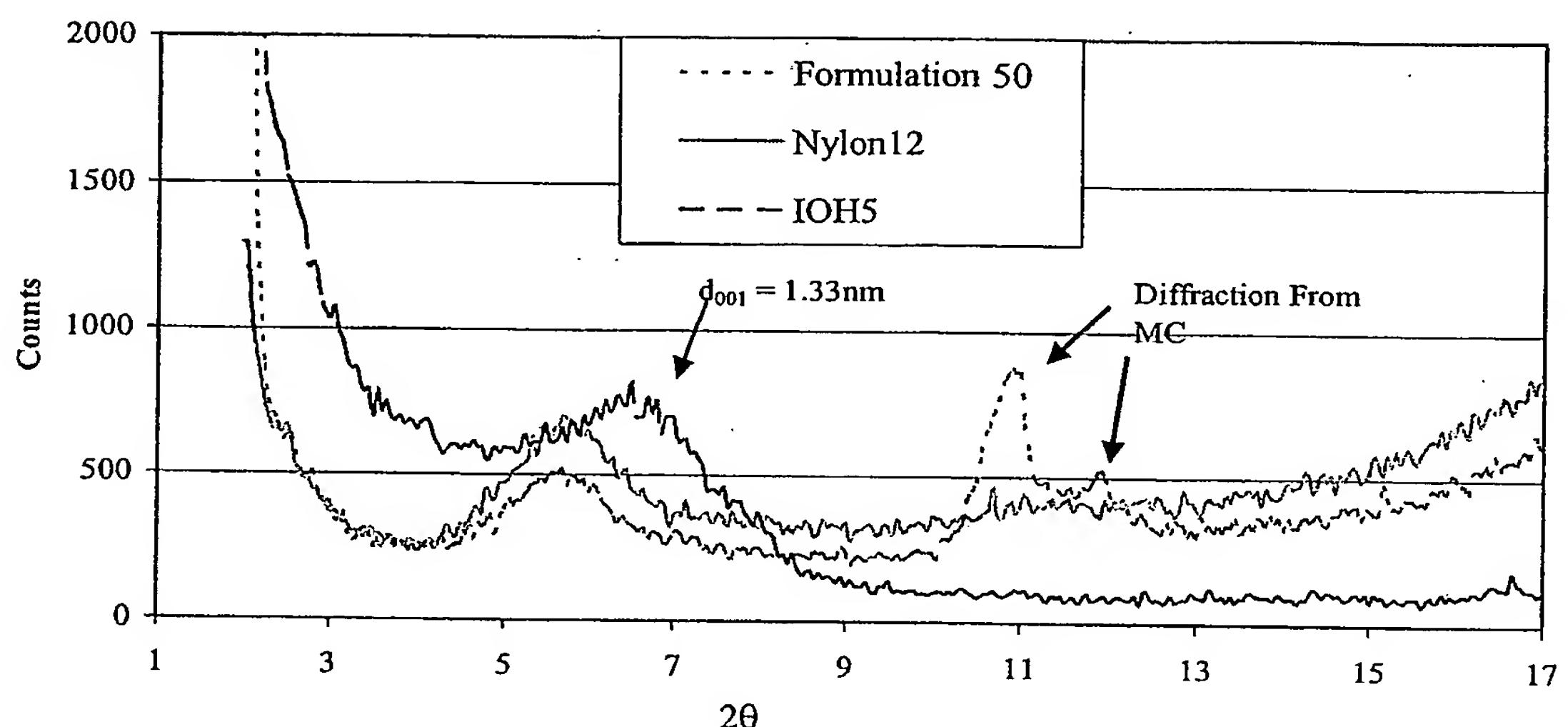


Figure 5

10/533579

WO 2004/039916

PCT/AU2003/001443

6 / 6

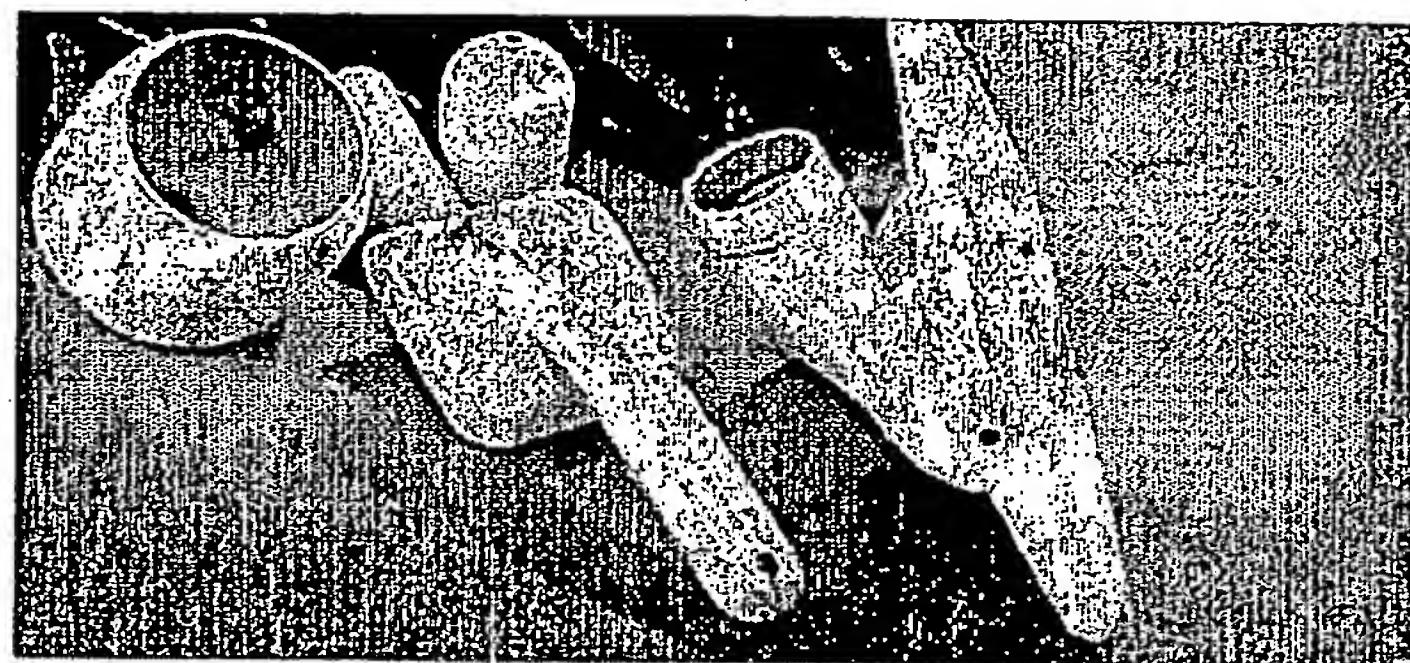


Figure 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU03/01443

A. CLASSIFICATION OF SUBJECT MATTER	
Int. Cl. ?: C09K 21/06, C01B 33/44, C08K 3/34	
According to International Patent Classification (IPC) or to both national classification and IPC	
B. FIELDS SEARCHED	
Minimum documentation searched (classification system followed by classification symbols)	
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched	
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) WPAT: IPC C09K 21/-, C08K 3/-; Keywords intercalated, onium, triazine, layered, melamine, and similar words	
C. DOCUMENTS CONSIDERED TO BE RELEVANT	
Category*	Citation of document, with indication, where appropriate, of the relevant passages
X	Derwent Abstract Accession No. 97-073190/07, Class A23 A85 A95, JP 08319418 A (SHOWA DENKO KK) 3 December 1996 Abstract
X	Derwent Abstract Accession No. 2002-599501/64, Class A18 E19 G02 J04 A21 A97, WO 200248248 A2 (AGROLINZ MELAMIN GMBH) 20 June 2002 Abstract
X	Derwent Abstract Accession No. 2002-443836/47, Class A96 A14 A17, WO 200216479 A1 (SEKISUI CHEM CO LTD) 28 February 2002 Abstract
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex	
<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier application or patent but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	
Date of the actual completion of the international search 28 November 2003	Date of mailing of the international search report - 5 DEC 2003
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized officer JAMES DZIEDZIC Telephone No : (02) 6283 2495

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU03/01443

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Patent Abstracts of Japan, JP 10-081510 A (SHOWA DENKO KK) 31 March 1998 Abstract	1-73
X	WO 9304117 A1 (ALLIED-SIGNAL INC) 4 March 1993 Whole document (Examples)	1-20,24- 36,42-51,63- 73
X	WO 9941299 A1 (SOLUTIA INC) 19 August 1999 Whole document (Examples)	1-20,26- 36,42-51,63- 73

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU03/01443

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
JP	8319418						
WO	0248248	CA	2431037	CZ	20031649	EE	200300287
		EP	1353994	HU	0302244	NO	20032712
WO	0216479	AU	80168/01	JP	2002138207	JP	2002138212
		JP	2003012942	JP	2003049136		
JP	10081510						
WO	9304117	CA	2115255	EP	0598836	US	5747560
		WO	9304118				
WO	9941299	AU	61623/98	BR	9815778	CA	2320988
		EP	1054922				
END OF ANNEX							

RECEIVED 02 MAY 2005

1/6

PCT REQUEST

FP18623

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

0	For receiving Office use only	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and "PCT International Application"	
0-4	Form - PCT/RO/101 PCT Request	
0-4-1	Prepared using PCT-EASY Version 2.92 (updated 01.07.2003)	
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant) Australian Patent Office (RO/AU)	
0-7	Applicant's or agent's file reference FP18623	
I	Title of invention FIRE RESISTANT MATERIAL	
II	Applicant	
II-1	This person is: applicant only	
II-2	Applicant for	
II-4	Name COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION	
II-5	Address: Limestone Avenue CAMPBELL, Australian Capital Territory 2612 Australia	
II-6	State of nationality AU	
II-7	State of residence AU	
III-1	Applicant and/or inventor	
III-1-1	This person is: applicant only	
III-1-2	Applicant for	
III-1-4	Name THE BOEING COMPANY	
III-1-5	Address: 7755 East Marginal Way South SEATTLE, WA 98108	
III-1-6	State of nationality United States of America	
III-1-7	State of residence US	

PCT REQUEST

2/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

III-2	Applicant and/or Inventor	
III-2-1	This person is:	applicant and inventor
III-2-2	Applicant for	US only
III-2-4	Name (LAST, First)	ANGLIN, Matthew, Allen
III-2-5	Address:	6403 Linden Avenue North SEATTLE, WA 98103
III-2-6	State of nationality	United States of America
III-2-7	State of residence	US
III-3	Applicant and/or Inventor	
III-3-1	This person is:	applicant and inventor
III-3-2	Applicant for	all designated States
III-3-4	Name (LAST, First)	BATEMAN, Stuart, Arthur
III-3-5	Address:	3 Wortley Avenue MOUNT WAVERLEY, Victoria 3149
III-3-6	State of nationality	Australia
III-3-7	State of residence	AU
III-4	Applicant and/or Inventor	
III-4-1	This person is:	applicant and inventor
III-4-2	Applicant for	US only
III-4-4	Name (LAST, First)	MACLEAN, Barbara, Lord
III-4-5	Address:	9824-NE 13th Street BELLEVUE, WA 98004
III-4-6	State of nationality	United States of America
III-4-7	State of residence	US
III-5	Applicant and/or Inventor	
III-5-1	This person is:	applicant and inventor
III-5-2	Applicant for	US only
III-5-4	Name (LAST, First)	PARTLETT, Matthew, Jon
III-5-5	Address:	1/69-71 Noble Street ALLAWAH, New South Wales 2218
III-5-6	State of nationality	Australia
III-5-7	State of residence	AU
III-6	Applicant and/or Inventor	
III-6-1	This person is:	applicant and inventor
III-6-2	Applicant for	US only
III-6-4	Name (LAST, First)	PETERSON, James, Macon
III-6-5	Address:	420 SW 183rd Street SEATTLE, WA 98166-39342
III-6-6	State of nationality	United States of America
III-6-7	State of residence	US

PCT REQUEST

3/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

III-7 Applicant and/or inventor		
III-7-1	This person is:	applicant and inventor
III-7-2	Applicant for	US only
III-7-4	Name (LAST, First)	WU, Dong, Yang
III-7-5	Address:	6 Arnott Court WHEELERS HILL, Victoria 3150 Australia
III-7-6	State of nationality	AU
III-7-7	State of residence	AU
IV-1 Agent or common representative; or address for correspondence The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: Name		agent
IV-1-1	Name	GRIFFITH HACK
IV-1-2	Address:	509 St Kilda Road MELBOURNE, Victoria 3004 Australia
IV-1-3	Telephone No.	+61 3 9243 8300
IV-1-4	Facsimile No.	+61 3 9243 8333
IV-1-5	e-mail	ghmelb@griffithhack.com.au
V Designation of States		
V-1 Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)		<p>AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZM ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT</p> <p>EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT</p> <p>EP: AT BE BG CH&LI CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL PT RO SE SI SK TR and any other State which is a Contracting State of the European Patent Convention and of the PCT</p> <p>OA: BF BJ CF CG CI CM GA GN GQ GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT</p>
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CO CR CU CZ DE DK DM DZ EC EE EG ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NI NO NZ OM PG PH PL PT RO RU SC SD SE SG SK SL SY TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW

PCT REQUEST

4/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

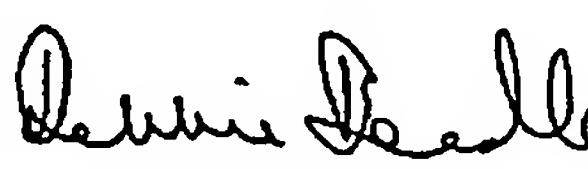
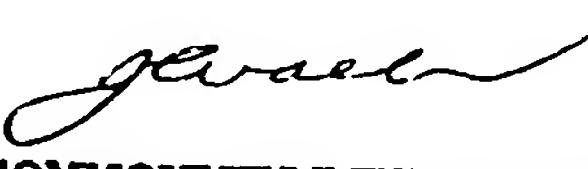
V-5	Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.		
V-6	Exclusion(s) from precautionary designations	NONE	
VI-1	Priority claim of earlier national application		
VI-1-1	Filing date	31 October 2002 (31.10.2002)	
VI-1-2	Number	2002952373	
VI-1-3	Country	AU	
VI-2	Priority document request The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1	
VII-1	International Searching Authority Chosen	Australian Patent Office (ISA/AU)	
VIII	Declarations	Number of declarations	
VIII-1	Declaration as to the identity of the inventor	-	
VIII-2	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	-	
VIII-3	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	-	
VIII-4	Declaration of inventorship (only for the purposes of the designation of the United States of America)	-	
VIII-5	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	-	
IX	Check list	number of sheets	electronic file(s) attached
IX-1	Request (including declaration sheets)	6	-
IX-2	Description	60	-
IX-3	Claims	11	-
IX-4	Abstract	1	EZABST00.TXT
IX-5	Drawings	6	-
IX-7	TOTAL	84	

PCT REQUEST

5/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18823

	Accompanying items	paper document(s) attached	electronic file(s) attached
IX-8	Fee calculation sheet	✓	-
IX-17	PCT-EASY diskette	-	Diskette
IX-19	Figure of the drawings which should accompany the abstract		
IX-20	Language of filing of the international application	English	
X-1	Signature of applicant, agent or common representative		
X-1-1	Name	GRIFFITH HACK	
X-1-2	Name of signatory	Debbie Beadle	
X-1-3	Capacity	Patent Attorney	
X-2	Signature of applicant, agent or common representative		
X-2-1	JOHN HERBERT WALKER Name Authorised Officer	COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION	
X-3	Signature of applicant, agent or common representative		
X-3-1	Name	THE BOEING COMPANY	
X-4	Signature of applicant, agent or common representative		
X-4-1	Name (LAST, First)	ANGLIN, Matthew, Allen	
X-5	Signature of applicant, agent or common representative		
X-5-1	Name (LAST, First)	BATEMAN, Stuart, Arthur	
X-6	Signature of applicant, agent or common representative		
X-6-1	Name (LAST, First)	MACLEAN, Barbara, Lord	
X-7	Signature of applicant, agent or common representative		
X-7-1	Name (LAST, First)	PARTLETT, Matthew, Jon	
X-8	Signature of applicant, agent or common representative		
X-8-1	Name (LAST, First)	PETERSON, James, Macon	
X-9	Signature of applicant, agent or common representative		
X-9-1	Name (LAST, First)	WU, Dong, Yang	

PCT REQUEST

6/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

FOR RECEIVING OFFICE USE ONLY

10-1	Date of actual receipt of the purported international application	
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
10-4	Date of timely receipt of the required corrections under PCT Article 11(2)	
10-5	International Searching Authority	ISA/AU
10-6	Transmittal of search copy delayed until search fee is paid	

FOR INTERNATIONAL BUREAU USE ONLY

11-1	Date of receipt of the record copy by the International Bureau	
------	--	--

PCT REQUEST

1/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

0	For receiving Office use only	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and "PCT International Application"	
0-4	Form - PCT/RO/101 PCT Request	
0-4-1	Prepared using	
0-5	Petition The undersigned requests that the present International application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	
0-7	Applicant's or agent's file reference	
I	Title of Invention	
II	Applicant	
II-1	This person is:	
II-2	Applicant for	
II-4	Name	
II-5	Address:	
II-6	State of nationality	
II-7	State of residence	
III-1	Applicant and/or inventor	
III-1-1	This person is:	
III-1-2	Applicant for	
III-1-4	Name	
III-1-5	Address:	
III-1-6	State of nationality	
III-1-7	State of residence	

PCT REQUEST

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

III-2	Applicant and/or Inventor	
III-2-1	This person is:	applicant and inventor
III-2-2	Applicant for	US only
III-2-4	Name (LAST, First)	ANGLIN, Matthew, Allen
III-2-5	Address:	6403 Linden Avenue North SEATTLE, WA 98103
III-2-6	State of nationality	United States of America
III-2-7	State of residence	US
III-3	Applicant and/or Inventor	
III-3-1	This person is:	applicant and inventor
III-3-2	Applicant for	all designated States
III-3-4	Name (LAST, First)	BATEMAN, Stuart, Arthur
III-3-5	Address:	3 Wortley Avenue MOUNT WAVERLEY, Victoria 3149
III-3-6	State of nationality	Australia
III-3-7	State of residence	AU
III-4	Applicant and/or Inventor	
III-4-1	This person is:	applicant and inventor
III-4-2	Applicant for	US only
III-4-4	Name (LAST, First)	MACLEAN, Barbara, Lord
III-4-5	Address:	9824-NE 13th Street BELLEVUE, WA 98004
III-4-6	State of nationality	United States of America
III-4-7	State of residence	US
III-5	Applicant and/or Inventor	
III-5-1	This person is:	applicant and inventor
III-5-2	Applicant for	US only
III-5-4	Name (LAST, First)	PARTLETT, Matthew, Jon
III-5-5	Address:	1/69-71 Noble Street ALLAWAH, New South Wales 2218
III-5-6	State of nationality	Australia
III-5-7	State of residence	AU
III-6	Applicant and/or Inventor	
III-6-1	This person is:	applicant and inventor
III-6-2	Applicant for	US only
III-6-4	Name (LAST, First)	PETERSON, James, Macon
III-6-5	Address:	420 SW 183rd Street SEATTLE, WA 98166-39342
III-6-6	State of nationality	United States of America
III-6-7	State of residence	US

PCT REQUEST

3/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

III-7 Applicant and/or Inventor		
III-7-1	This person is:	
III-7-2	Applicant for	
III-7-4	Name (LAST, First)	
III-7-5	Address:	
III-7-6	State of nationality	
III-7-7	State of residence	
IV-1	Agent or common representative; or address for correspondence The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as:	
IV-1-1	Name	
IV-1-2	Address:	
IV-1-3	Telephone No.	
IV-1-4	Facsimile No.	
IV-1-5	e-mail	
V	Designation of States	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	

applicant and inventor
US only
WU, Dong, Yang
6 Arnott Court
WHEELERS HILL, Victoria 3150
Australia
AU
AU

agent
GRIFFITH HACK
509 St Kilda Road
MELBOURNE, Victoria 3004
Australia
+61 3 9243 8300
+61 3 9243 8333
ghmelb@griffithhack.com.au

AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZM
ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT
EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
EP: AT BE BG CH&LI CY CZ DE DK EE ES FI
FR GB GR HU IE IT LU MC NL PT RO SE SI
SK TR and any other State which is a Contracting State of the European Patent Convention and of the PCT
OA: BF BJ CF CG CI CM GA GN GQ GW ML MR
NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT

AE AG AL AM AT AU AZ BA BB BG BR BY BZ
CA CH&LI CN CO CR CU CZ DE DK DM DZ EC
EE EG ES FI GB GD GE GH GM HR HU ID IL
IN IS JP KE KG KP KR KZ LC LK LR LS LT
LU LV MA MD MG MK MN MW MX MZ NI NO NZ
OM PG PH PL PT RO RU SC SD SE SG SK SL
SY TJ TM TN TR TT TZ UA UG US UZ VC VN
YU ZA ZM ZW

PCT REQUEST

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

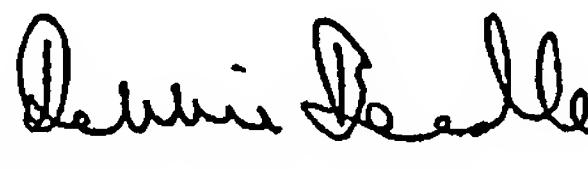
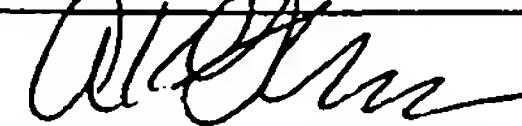
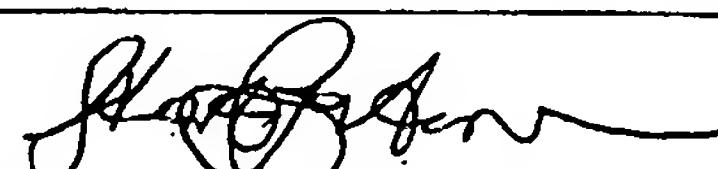
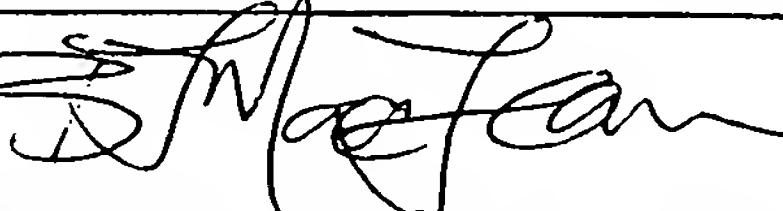
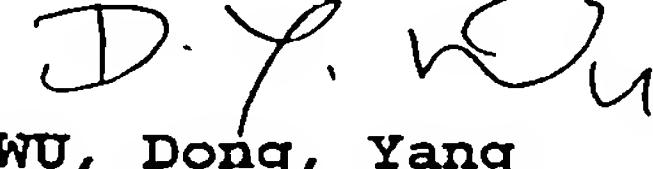
V-5	Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.		
V-6	Exclusion(s) from precautionary designations	NONE	
VI-1	Priority claim of earlier national application Filing date	31 October 2002 (31.10.2002)	
VI-1-1	Number	2002952373	
VI-1-2	Country	AU	
VI-2	Priority document request The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1	
VII-1	International Searching Authority Chosen	Australian Patent Office (ISA/AU)	
VIII	Declarations	Number of declarations	
VIII-1	Declaration as to the identity of the inventor	-	
VIII-2	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	-	
VIII-3	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	-	
VIII-4	Declaration of inventorship (only for the purposes of the designation of the United States of America)	-	
VIII-5	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	-	
IX	Check list	number of sheets	electronic file(s) attached
IX-1	Request (including declaration sheets)	6	-
IX-2	Description	60	-
IX-3	Claims	11	-
IX-4	Abstract	1	EZABST00.TXT
IX-5	Drawings	6	-
IX-7	TOTAL	84	

PCT REQUEST

5/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

	Accompanying items	paper document(s) attached	electronic file(s) attached
IX-8	Fee calculation sheet	✓	-
IX-17	PCT-EASY diskette	-	Diskette
IX-19	Figure of the drawings which should accompany the abstract		
IX-20	Language of filing of the international application	English	
X-1	Signature of applicant, agent or common representative		
X-1-1	Name	GRIFFITH HACK	
X-1-2	Name of signatory	Debbie Beadle	
X-1-3	Capacity	Patent Attorney	
X-2	Signature of applicant, agent or common representative		
X-2-1	Name	COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION	
X-3	Signature of applicant, agent or common representative		
X-3-1	Name	Ann K. Galbraith, Attorney THE BOEING COMPANY	
X-4	Signature of applicant, agent or common representative		
X-4-1	Name (LAST, First)	ANGLIN, Matthew, Allen	
X-5	Signature of applicant, agent or common representative		
X-5-1	Name (LAST, First)	BATEMAN, Stuart, Arthur	
X-6	Signature of applicant, agent or common representative		
X-6-1	Name (LAST, First)	MACLEAN, Barbara, Lord	
X-7	Signature of applicant, agent or common representative		
X-7-1	Name (LAST, First)	PARTLETT, Matthew, Jon	
X-8	Signature of applicant, agent or common representative		
X-8-1	Name (LAST, First)	PETERSON, James, Macon	
X-9	Signature of applicant, agent or common representative		
X-9-1	Name (LAST, First)		
		WU, Dong, Yang	

PCT REQUEST

6/6

FP18623

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FOR RECEIVING OFFICE USE ONLY

10-1	Date of actual receipt of the purported international application	
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
10-4	Date of timely receipt of the required corrections under PCT Article 11(2)	
10-5	International Searching Authority	ISA/AU
10-6	Transmittal of search copy delayed until search fee is paid	

FOR INTERNATIONAL BUREAU USE ONLY

11-1	Date of receipt of the record copy by the International Bureau	
------	--	--

PCT REQUEST

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

0	For receiving Office use only	
0-1	International Application No.	
0-2	International Filing Date	
0-3	Name of receiving Office and "PCT International Application"	
0-4	Form - PCT/RO/101 PCT Request	
0-4-1	Prepared using	
PCT-EASY Version 2.92 (updated 01.07.2003)		
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	
Australian Patent Office (RO/AU)		
0-7	Applicant's or agent's file reference	
FP18623		
I	Title of invention	
FIRE RESISTANT MATERIAL		
II	Applicant	
II-1	This person is:	
II-2	Applicant for	
II-4	Name	
II-5	Address:	
all designated States except US COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION Limestone Avenue CAMPBELL, Australian Capital Territory 2612 Australia		
II-6	State of nationality	
II-7	State of residence	
AU AU		
III-1	Applicant and/or Inventor	
III-1-1	This person is:	
III-1-2	Applicant for	
III-1-4	Name	
III-1-5	Address:	
all designated States except US THE BOEING COMPANY 7755 East Marginal Way South SEATTLE, WA 98108 United States of America		
III-1-6	State of nationality	
III-1-7	State of residence	
US US		

PCT REQUEST

2/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

III-2	Applicant and/or inventor	
III-2-1	This person is:	applicant and inventor
III-2-2	Applicant for	US only
III-2-4	Name (LAST, First)	ANGLIN, Matthew, Allen
III-2-5	Address:	6403 Linden Avenue North SEATTLE, WA 98103
III-2-6	State of nationality	United States of America
III-2-7	State of residence	US
III-3	Applicant and/or inventor	
III-3-1	This person is:	applicant and inventor
III-3-2	Applicant for	all designated States
III-3-4	Name (LAST, First)	BATEMAN, Stuart, Arthur
III-3-5	Address:	3 Wortley Avenue MOUNT WAVERLEY, Victoria 3149
III-3-6	State of nationality	Australia
III-3-7	State of residence	AU
III-4	Applicant and/or inventor	
III-4-1	This person is:	applicant and inventor
III-4-2	Applicant for	US only
III-4-4	Name (LAST, First)	MACLEAN, Barbara, Lord
III-4-5	Address:	9824-NE 13th Street BELLEVUE, WA 98004
III-4-6	State of nationality	United States of America
III-4-7	State of residence	US
III-5	Applicant and/or inventor	
III-5-1	This person is:	applicant and inventor
III-5-2	Applicant for	US only
III-5-4	Name (LAST, First)	PARTLETT, Matthew, Jon
III-5-5	Address:	1/69-71 Noble Street ALLAWAH, New South Wales 2218
III-5-6	State of nationality	Australia
III-5-7	State of residence	AU
III-6	Applicant and/or inventor	
III-6-1	This person is:	applicant and inventor
III-6-2	Applicant for	US only
III-6-4	Name (LAST, First)	PETERSON, James, Macon
III-6-5	Address:	420 SW 183rd Street SEATTLE, WA 98166-39342
III-6-6	State of nationality	United States of America
III-6-7	State of residence	US

PCT REQUEST

3/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

III-7	Applicant and/or Inventor	
III-7-1	This person is:	
III-7-2	Applicant for	
III-7-4	Name (LAST, First)	
III-7-5	Address:	
III-7-6	State of nationality	
III-7-7	State of residence	
IV-1	Agent or common representative; or address for correspondence The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: Name Address:	
IV-1-1	Name GRIFFITH HACK	
IV-1-2	Address: 509 St Kilda Road MELBOURNE, Victoria 3004 Australia +61 3 9243 8300 +61 3 9243 8333 ghmelb@griffithhack.com.au	
IV-1-3	Telephone No.	
IV-1-4	Facsimile No.	
IV-1-5	e-mail	
V	Designation of States	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZM ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE BG CH&LI CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL PT RO SE SI SK TR and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GQ GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT	
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CO CR CU CZ DE DK DM DZ EC EE EG ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NI NO NZ OM PG PH PL PT RO RU SC SD SE SG SK SL SY TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW	

PCT REQUEST

4/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

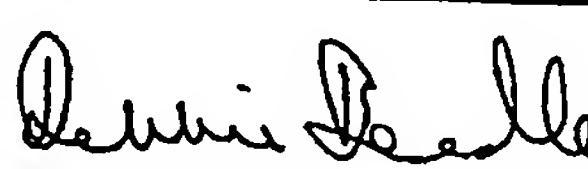
V-5	Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.	
V-6	Exclusion(s) from precautionary designations	NONE
VI-1	Priority claim of earlier national application	
VI-1-1	Filing date	31 October 2002 (31.10.2002)
VI-1-2	Number	2002952373
VI-1-3	Country	AU
VI-2	Priority document request The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) identified above as item(s):	VI-1
VII-1	International Searching Authority Chosen	Australian Patent Office (ISA/AU)
VIII	Declarations	Number of declarations
VIII-1	Declaration as to the identity of the inventor	-
VIII-2	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	-
VIII-3	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	-
VIII-4	Declaration of inventorship (only for the purposes of the designation of the United States of America)	-
VIII-5	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	-
IX	Check list	
IX-1	Request (including declaration sheets)	number of sheets
IX-2	Description	6
IX-3	Claims	60
IX-4	Abstract	11
IX-5	Drawings	1
IX-6		EZABST00.TXT
IX-7	TOTAL	84

PCT REQUEST

5/6

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FP18623

	Accompanying items	paper document(s) attached	electronic file(s) attached
IX-8	Fee calculation sheet	✓	-
IX-17	PCT-EASY diskette	-	Diskette
IX-19	Figure of the drawings which should accompany the abstract		
IX-20	Language of filing of the International application	English	
X-1	Signature of applicant, agent or common representative	 DEBBIE BEADLE	
X-1-1	Name	GRIFFITH HACK	
X-1-2	Name of signatory	Debbie Beadle	
X-1-3	Capacity	Patent Attorney	
X-2	Signature of applicant, agent or common representative		
X-2-1	Name	COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION	
X-3	Signature of applicant, agent or common representative		
X-3-1	Name	THE BOEING COMPANY	
X-4	Signature of applicant, agent or common representative		
X-4-1	Name (LAST, First)	ANGLIN, Matthew, Allen	
X-5	Signature of applicant, agent or common representative		
X-5-1	Name (LAST, First)	BATEMAN, Stuart, Arthur	
X-6	Signature of applicant, agent or common representative		
X-6-1	Name (LAST, First)	MACLEAN, Barbara, Lord	
X-7	Signature of applicant, agent or common representative	 PARTLETT, Matthew, Jon	
X-7-1	Name (LAST, First)		
X-8	Signature of applicant, agent or common representative		
X-8-1	Name (LAST, First)	PETERSON, James, Macon	
X-9	Signature of applicant, agent or common representative		
X-9-1	Name (LAST, First)	WU, Dong, Yang	

PCT REQUEST

6/6

FP18623

Original (for SUBMISSION) - printed on 31.10.2003 11:37:57 AM

FOR RECEIVING OFFICE USE ONLY

10-1	Date of actual receipt of the purported international application	
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
10-4	Date of timely receipt of the required corrections under PCT Article 11(2)	
10-5	International Searching Authority	ISA/AU
10-6	Transmittal of search copy delayed until search fee is paid	

FOR INTERNATIONAL BUREAU USE ONLY

11-1	Date of receipt of the record copy by the International Bureau	
------	--	--

The demand must be filed directly with the International Preliminary Examining Authority or, if more Authorities are competent, with the one chosen by the applicant. The full name and letter code of that Authority may be indicated by the applicant on the line below:

IPEA/_____

PCT
DEMAND

CHAPTER II

under Article 31 of the Patent Cooperation Treaty:

The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation

For international Preliminary Examining Authority use only

Identification of IPEA	Date of receipt of DEMAND
------------------------	---------------------------

Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION		Applicant's or agents file reference DAB:FP18623
International application No. PCT/AU03/01443	International filing date (day/month/year) 31 October 2003	(Earliest) Priority date (day/month/year) 31 October 2002

Title of the invention FIRE RESISTANT MATERIAL	
---	--

Box No. II APPLICANT(S)		Telephone No.
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country) COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION LIMESTONE AVENUE CAMPBELL ACT 2612 AUSTRALIA		Facsimile No.
		Teleprinter No.
		Applicant's Registration No. with the office

State (that is, country) of nationality: AUSTRALIA	State (that is, country) of residence: AUSTRALIA
---	---

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country) THE BOEING COMPANY 7755 EAST MARGINAL WAY SOUTH SEATTLE WA 98108 UNITED STATES	
--	--

State (that is, country) of nationality: UNITED STATES	State (that is, country) of residence: UNITED STATES
---	---

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country) ANGLIN, Matthew, Allen 6403 Linden Avenue North SEATTLE, WA 98103 UNITED STATES	
---	--

State (that is, country) of nationality: UNITED STATES	State (that is, country) of residence: UNITED STATES
---	---

Further applicants are indicated on a continuation sheet.

Continuation of Box No. II APPLICANT(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

BATEMAN, Stuart, Arthur
3 Wortley Avenue
MOUNT WAVERLEY, VIC 3149
AUSTRALIA

State (that is, country) of nationality:
AUSTRALIA

State (that is, country) of residence:
AUSTRALIA

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

MACLEAN, Barbara, Lord
9824-NE 13th Street
BELLEVUE, WA 98004
UNITED STATES

State (that is, country) of nationality:
UNITED STATES

State (that is, country) of residence:
UNITED STATES

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

PARTLETT, Matthew, Jon
1/69-71 Noble Street
ALLAWAH, NSW 2218
AUSTRALIA

State (that is, country) of nationality:
AUSTRALIA

State (that is, country) of residence:
AUSTRALIA

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

PETERSON, James, Macon
420 SW 183rd Street
SEATTLE, WA 98166-39342
UNITED STATES

State (that is, country) of nationality:
UNITED STATES

State (that is, country) of residence:
UNITED STATES

Further applicants are indicated on a continuation sheet.

Continuation of Box No. II APPLICANT(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

WU, Dong, Yang
6 Arnott Court
WHEELERS HILL, VIC 3150
AUSTRALIA

State (that is, country) of nationality:
AUSTRALIA

State (that is, country) of residence:
AUSTRALIA

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

State (that is, country) of nationality:

State (that is, country) of residence:

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

State (that is, country) of nationality:

State (that is, country) of residence:

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country)

State (that is, country) of nationality:

State (that is, country) of residence:

Further applicants are indicated on a continuation sheet.

Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The following person is agent common representative
 and has been appointed earlier and represents the applicant(s) also for international preliminary examination.
 is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked
 is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.

Name and address: (Family name followed by given name; for a legal entity, full official designation.
 The address must include postal code and name of country)

Debbie Beadle
 Griffith Hack
 509 ST KILDA ROAD
 MELBOURNE VIC 3004

Telephone No.	+61 3 9243 8300
Facsimile No.	+61 3 9243 8333
Teleprinter No.	
Agent's Registration No. with the office	

Address for correspondence: Mark this checkbox where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION**Statement concerning amendments***

1. The applicant wishes the international preliminary examination to start on the basis of:

The international application as originally filed

the description as originally filed
 as amended under Article 34

the claims as originally filed
 as amended under Article 19 (together with any accompanying statement)
 as amended under Article 34

the drawings as originally filed
 as amended under Article 34
2. The applicant wishes any amendment to the claim under Article 19 to be considered reversed.
3. The applicant wishes the start of the international preliminary examination to be postponed until the expiration of the applicable time limit under Rule 69.1(d).
4. The applicant expressly wishes the international preliminary examination to start earlier than at the expiration of the applicable time limit under Rule 54bis.1(a).

* Where no checkbox is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purpose of international preliminary examination: ENGLISH

which is the language in which the international application is filed

which is the language of a translation furnished for the purposes in international search

which is the language of publication of the international application

which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

Box No. V ELECTION OF STATES

The filing of this demand constitutes the election of all Contracting States which are designated and are bound by Chapter II of the PCT.

Box no. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

1. translation of international application:	sheets
2. amendments under Article 34:	sheets
3. copy (or where required, translation) of amendments under Article 19:	sheets
4. copy (or where required, translation) of statement under Article 19:	sheets
5. letter:	sheets
6. other (specify):	sheets

For International Preliminary Examining Authority use only

received	not received
----------	--------------

The demand is accompanied by the item(s) marked below:

1. <input checked="" type="checkbox"/> fee calculation sheet	5. <input type="checkbox"/> statement explaining lack of signature
2. <input type="checkbox"/> separate signed power of attorney	6. <input type="checkbox"/> sequence listing in computer readable form
3. <input type="checkbox"/> original general power of attorney	7. <input type="checkbox"/> tables in computer readable form related to a sequence listing
4. <input type="checkbox"/> copy of general power of attorney; reference number, if any:	8. <input type="checkbox"/> other (specify):

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand)



28/5/04

Signature

Date

DEBBIE BEADLE
GRIFFITH HACK
509 ST KILDA ROAD,
MELBOURNE VICTORIA 3004
AUSTRALIA

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND:		
2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):		
3. <input type="checkbox"/> The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply	6. <input type="checkbox"/> The date of receipt of the demand is AFTER the expiration of the time limit under Rule 54bis.1(a) and item 7 or 8, below, does not apply.	
<input type="checkbox"/> The applicant has been informed accordingly	7. <input type="checkbox"/> The date of receipt of the demand is WITHIN the time limit under Rule 54bis.1(a) as extended by virtue of Rule 80.5.	
4. <input type="checkbox"/> The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of rule 80.5	8. <input type="checkbox"/> Although the date of receipt of the demand is after the expiration of the time limit under Rule 54bis.1(a), the delay in arrival is EXCUSED pursuant to Rule 82.	
5. <input type="checkbox"/> Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.		

For International Bureau use only

Demand received from IPEA on:

FEE CALCULATION SHEET

Annex to the Demand for international preliminary examination

International application No. PCT/AU03/01443	For International Preliminary Examining Authority use only
Applicant's or Agent's file reference DAB:FP18623	Date stamp of the IPEA
Applicant COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION THE BOEING COMPANY	
Calculation of prescribed fees	
1. Preliminary examination fee	\$550
2. Handling fee (<i>Applicants from certain states are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25 of the handling fee.</i>)	\$218
3. Total prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box	\$768
	TOTAL
Mode of Payment	
<input type="checkbox"/> authorisation to charge deposit account with the IPEA (see below)	<input type="checkbox"/> cash
<input type="checkbox"/> cheque	<input type="checkbox"/> revenue stamps
<input type="checkbox"/> postal money order	<input type="checkbox"/> coupons
<input type="checkbox"/> bank draft	<input type="checkbox"/> other (<i>specify</i>): _____
Authorisation to Charge (or Credit) Deposit Account (<i>This mode of payment may not be available at all IPEA's</i>)	
<input type="checkbox"/> Authorisation to charge the total fees indicated above	IPEA/ _____
<input type="checkbox"/> (<i>this check box may be marked only if the conditions for deposit accounts of the IPEA so permit</i>) Authorisation to charge any deficiency or credit any overpayment in the total fees indicated above	Deposit Account Number _____
	Date _____
	Name _____
	Signature _____

From the:
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

PATENT-COOPERATION TREATY

To:

Griffith Hack
GPO Box 1285K
MELBOURNE VIC 3001

GRIFFITH HACK	
- 9 JUN 2004	
1.....	<i>JCH</i>
2.....	<i>DAB</i>
3.....	

DIARIED

ID. 359387

PCT

WRITTEN OPINION
(PCT Rule 66)Date of mailing
(day/month/year)

09 JUN 2004

REPLY DUE

within **TWO MONTHS**
from the above date of mailingApplicant's or agent's file reference
DAB:ST:FP18623International Application No.
PCT/AU2003/001443

International Filing Date (day/month/year)

31 October 2003

Priority Date (day/month/year)

31 October 2002

International Patent Classification (IPC) or both national classification and IPC
Int. Cl. 7 C09K 21/06, C01B 33/44, C08K 3/34

Applicant

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al

BOEING

1. This written opinion is the **first** drawn by this International Preliminary Examining Authority.
2. This opinion contains indications relating to the following items:

I Basis of the opinion

II Priority

III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

IV Lack of unity of invention

V Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

VI Certain documents cited

VII Certain defects in the international application

VIII Certain observations on the international application

3. The **FINAL DATE** by which the international preliminary examination report must be established according to Rule 69.2 is:
28 February 2005

The applicant is hereby invited to reply to this opinion.

When? See the **Reply Due** date indicated above. However, the Australian Patent Office will not establish the Report before the earlier of (i) a response being filed, or (ii) one month before the **Final Date** by which the international preliminary examination report must be established. The Report will take into account any response (including amendments) filed before the Report is established.

If no response is filed by 1 month before the **Final Date**, the international preliminary examination report will be established on the basis of this opinion.

Applicants wishing to have the benefit of a further opinion (if needed) before the report is established should ensure that a response is filed at least 3 months before the **Final Date** by which the international preliminary examination report must be established.

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4. For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4bis. For an informal communication with the examiner, see Rule 66.6.

Name and mailing address of the IPEA/AU
AUSTRALIAN PATENT OFFICE
PO BOX 200, WODEN ACT 2606, AUSTRALIA
E-mail address: pct@ipaaustralia.gov.au
Facsimile No. (02) 6285 3929

Authorized Officer

JAMES DZIEDZIC

Telephone No. (02) 6283 2495

WRITTEN OPINION

International application No.

PCT/AU2003/001443

I. Basis of the opinion

1. With regard to the elements of the international application:*

the international application as originally filed.

the description, pages **Abstract**, as originally filed,
pages , filed with the demand,
pages **1-60**, received on **12 February 2004** with the letter of **12 February 2004**

the claims, pages , as originally filed,
pages , as amended under Article 19;
pages , filed with the demand,
pages **61-71**, received on **12 February 2004** with the letter of **12 February 2004**

the drawings, pages **1/6 - 6/6**, as originally filed,
pages , filed with the demand,
pages , received on with the letter of

the sequence listing part of the description:
pages , as originally filed
pages , filed with the demand
pages , received on with the letter of

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language which is:

the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

the language of publication of the international application (under Rule 48.3(b)).

the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:

contained in the international application in printed form.

filed together with the international application in computer readable form.

furnished subsequently to this Authority in written form.

furnished subsequently to this Authority in computer readable form.

The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

the description, pages

the claims, Nos.

the drawings, sheets/fig.

5. This opinion has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed"

WRITTEN OPINION

International application No.

PCT/AU2003/001443

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	YES
	Claims 1-73	NO
Inventive step (IS)	Claims	YES
	Claims 1-73	NO
Industrial applicability (IA)	Claims 1-73	YES
	Claims	NO

2. Citations and explanations

Citations

- D1. WO 2002/048248 A2
- D2. JP 08-319418 A
- D3. JP 2002-138212 A (SEKISUI CHEM CO LTD) 14 May 2002 (Equivalent of WO 2002/16479 A1).
- D4. JP 10-081510 A
- D5. WO 1993/004117 A1
- D6. WO 1999/041299 A1

NOVELTY (N) & INVENTIVE STEP (IS) Claims 1-73

D1 discloses (see claims 1, 2, 7) a flame retardant composition comprising an expandable layered inorganic component (eg montmorillonite) and an ionic organic component (eg organic salts of melamine).

D2 discloses (see paragraphs 10-14) a flame retardant composition comprising a polyamide resin, an expandable layered inorganic component (eg montmorillonite) and an ionic organic component (eg isocyanuric acid and melamine).

D3 discloses (see Example 3) a flame retardant composition comprising a polypropylene resin, an expandable layered inorganic component (eg fluorine mica) and an ionic organic component (eg N-polyoxyethylene-N-lauryl-N and N-dimethylammonium).

D4 discloses (see Examples 1-17) a flame retardant composition comprising polyamide resin (eg Nylon 66 PA-2), an expandable layered inorganic component (eg fluorine synthetic mica SC-3) and an ionic organic component (eg cyanuric-acid melamine SC-3).

D5 discloses (see Example 1) a polymeric nanocomposite composition comprising polyamide resin (eg Nylon 6), an expandable layered inorganic component (eg montmorillonite) and an ionic organic component (eg dipentyl ammonium chloride).

D6 discloses (see Claims 1, 2, 4, 10) a polymeric nanocomposite composition comprising polyamide resin (eg Nylon 6), an expandable layered inorganic component (eg montmorillonite) and an ionic organic component (eg quaternary ammonium ions).

Therefore, it is considered that the claimed invention is not novel and not inventive in light of the disclosure contained in documents D1 to D6.



The Commissioner of Patents

25 January 2005

COPY

Madam

IN THE MATTER OF International Patent Application No. PCT/AU2003/001443
in the name of

**COMMONWEALTH SCIENTIFIC AND INDUSTRIAL
RESEARCH ORGANISATION and
THE BOEING COMPANY**

Entitled FIRE RESISTANT MATERIAL

Our Ref: DAB:AMM:FP18623

We refer to the Written Opinion dated 9 June 2004 and enclose new pages 6, 9, 61-71 to replace pages 6, 9, 61- 71 at present on file. Original claims 1, 2 and 19 have now been combined in order to distinguish the present invention from documents D1 to D6.

It is respectfully requested that the novelty and inventive step objections raised under Section V of the Written Opinion now be withdrawn.

Yours faithfully

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU03/01443

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. 7: C09K 21/06, C01B 33/44, C08K 3/34

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
WPAT: IPC C09K 21/-, C08K 3/-; Keywords intercalated, onium, triazine, layered, melamine, and similar words

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Derwent Abstract Accession No. 97-073190/07, Class A23 A85 A95, JP 08319418 A (SHOWA DENKO KK) 3 December 1996 Abstract	1-14,19- 23,31- 36,38,42- 54,56-73
X	Derwent Abstract Accession No. 2002-599501/64, Class A18 E19 G02 J04 A21 A97, WO 200248248 A2 (AGROLINZ MELAMIN GMBH) 20 June 2002 Abstract	1-27,31- 40,42-73
X	Derwent Abstract Accession No. 2002-443836/47, Class A96 A14 A17, WO 200216479 A1 (SEKISUI CHEM CO LTD) 28 February 2002 Abstract	1-16,19- 22,31- 36,38,41- 44,63-73

 Further documents are listed in the continuation of Box C See patent family annex

* Special categories of cited documents:	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search
28 November 2003

Date of mailing of the international search report

- 5 DEC 2003

Name and mailing address of the ISA/AU
AUSTRALIAN PATENT OFFICE
PO BOX 200, WODEN ACT 2606, AUSTRALIA
E-mail address: pct@ipaaustralia.gov.au
Facsimile No. (02) 6285 3929

Authorized officer

JAMES DZIEDZIC

Telephone No : (02) 6283 2495

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU03/01443

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Patent Abstracts of Japan, JP 10-081510 A (SHOWA DENKO KK) 31 March 1998 Abstract	1-73
X	WO 9304117 A1 (ALLIED-SIGNAL INC) 4 March 1993 Whole document (Examples)	1-20,24-36,42-51,63-73
X	WO 9941299 A1 (SOLUTIA INC) 19 August 1999 Whole document (Examples)	1-20,26-36,42-51,63-73

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU03/01443

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
JP	8319418						
WO	0248248	CA	2431037	CZ	20031649	EE	200300287
		EP	1353994	HU	0302244	NO	20032712
WO	0216479	AU	80168/01	JP	2002138207	JP	2002138212
		JP	2003012942	JP	2003049136		
JP	10081510						
WO	9304117	CA	2115255	EP	0598836	US	5747560
		WO	9304118				
WO	9941299	AU	61623/98	BR	9815778	CA	2320988
		EP	1054922				

END OF ANNEX

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference FP18623	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416).
International Application No. PCT/AU2003/001443	International Filing Date (day/month/year) 31 October 2003	Priority Date (day/month/year) 31 October 2002
International Patent Classification (IPC) or national classification and IPC Int. Cl. 7 C09K 21/06, C01B 33/44, C08K 3/34		
Applicant COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 3 sheets, including this cover sheet.

This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 13 sheet(s).

3. This report contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

Date of submission of the demand 28 May 2004	Date of completion of the report 22 February 2005
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer GAYE HOROBIN Telephone No. (02) 6283 2069

I. Basis of the report

1. With regard to the elements of the international application:*

 the international application as originally filed. the description, pages 1-5,7,8,10-60, as originally filed,

pages , filed with the demand,

pages 6,9, received on 25 January 2005 with the letter of 25 January 2005

 the claims, pages , as originally filed,

pages , as amended (together with any statement) under Article 19,

pages , filed with the demand,

pages 61-71, received on 25 January 2005 with the letter of 25 January 2005

 the drawings, pages 1/6-6/6, as originally filed,

pages , filed with the demand,

pages , received on with the letter of

 the sequence listing part of the description:

pages , as originally filed

pages , filed with the demand

pages , received on with the letter of

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language which is:

 the language of a translation furnished for the purposes of international search (under Rule 23.1(b)). the language of publication of the international application (under Rule 48.3(b)). the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

 contained in the international application in written form. filed together with the international application in computer readable form. furnished subsequently to this Authority in written form. furnished subsequently to this Authority in computer readable form. The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished. The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished4. The amendments have resulted in the cancellation of: the description, pages the claims, Nos. the drawings, sheets/fig.5. This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

** Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. Statement**

Novelty (N)	Claims 1-71	YES
	Claims	NO
Inventive step (IS)	Claims 1-71	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-71	YES
	Claims	NO

2. Citations and explanations (Rule 70.7)**NOVELTY(N), INVENTIVE STEP(IS)**

No citation or obvious combination of citations discloses the combination of at least one ionic organic compound and one or more neutral organic compounds intercalated between the layers of an expandable or swellable layered inorganic component.

Accordingly, there is a need for the development of new flame retarding systems which both meet the performance criteria associated with specific applications and address the above concerns.

5

SUMMARY OF THE INVENTION

According to one aspect of the present invention there is provided an inorganic-organic hybrid (IOH) which comprises:

10

(i) an expandable or swellable layered inorganic component; and

(ii) an organic component including at least one ionic organic component and one or more neutral organic components which are intercalated between and/or

15

associated with the layer(s) of the inorganic component,

the ionic or neutral organic components being capable of decomposing or subliming endothermically, and/or releasing volatiles with low combustibility on decomposition and/or inducing charring of organic species

20

during thermal decomposition or combustion.

25

According to another aspect of the present invention there is provided a method for the preparation of the IOH defined above which comprises mixing components (i) and (ii) defined above or constituents thereof in one or more steps.

The present invention also provides the use of the IOH defined above as a fire resistant material.

30

According to a further aspect of the present invention there is provided a fire resistant formulation which comprises:

(i) the IOH defined above; and

(ii) one or more flame retardants.

35

According to a still further aspect of the present invention there is provided a method for the preparation of the fire resistant formulation defined above which comprises mixing components (i) and (ii) as defined above or constituents thereof in one or more steps.

40

The present invention also provides a polyamide fire resistant formulation which comprises either:

- 9 -

of the inorganic component and/or one or more coupling reagents.

The term "associated with" is used herein in its broadest sense and refers to the neutral organic component 5 being attached to the layer(s) of the inorganic component, for example, by secondary bonding interactions, such as, Van der Waals interactions or hydrogen bonding or trapped by steric limitation.

Suitable examples of ionic species include those 10 that contain onium ions such as ammonium (primary, secondary, tertiary and quaternary), phosphonium or sulfonium derivatives of aliphatic, aromatic or aryl-aliphatic amines, phosphines and sulfides.

Such compounds may be prepared by any method 15 known to those skilled in the art. For example, salts prepared by acid-base type reactions with mineral or organic acids including hydrochloric, sulfuric, nitric, phosphoric, acetic and formic acids, by Lewis-acid - Lewis-base type reactions or by reaction with alkyl 20 halides to form quaternary salts for example using Menschutkin type methodology.

Ionic or neutral compounds which are known to decompose or sublime endothermically, and/or which release volatiles with low combustibility on decomposition and/or 25 induce charring of organic species during thermal decomposition or combustion are used in the IOH of the present invention.

Suitable species include neutral or ionic 30 derivatives of nitrogen based molecules, such as, triazine based species, for example, melamine, triphenyl melamine, melam (1,3,5-triazine-2,4,6-triamine-n-(4,6-diamino-1,3,5-triazine-yl)), melem ((-2,5,8-triamino-1,3,4,6,7,9,9b-heptaazaphenalene)), melon (poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalene-2,5-diyl)imino}), bis and 35 triaziridinyltriazine, trimethylsilyltriazine, melamine cyanurate, melamine phthalate, melamine phosphate, melamine phosphite, melamine phthalimide, dimelamine phosphate, phosphazines and/or low molecular weight

- 61 -

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An inorganic-organic hybrid (IOH) which comprises:
 - 5 (i) an expandable or swellable layered inorganic component; and
 - (ii) an organic component including at least one ionic organic component.
- 10 2. An IOH according to claim 1, in which the organic component also includes one or more neutral organic components which are intercalated between and/or associated with the layer(s) of the inorganic component.
- 15 3. An IOH according to claim 1 or claim 2, in which the inorganic component is rendered positively or negatively charged due to isomorphic substitution of elements within the layers.
- 20 4. An IOH according to any one of claims 1 to 3, in which the inorganic component is selected from a 1:1 layered silicate structure, a 2:1 layered silicate structure, a double hydroxide of the general formula $Mg_6Al_{3.4}(OH)_{18.8}(CO_3)_{1.7} \cdot H_2O$ and a synthetically prepared layered material.
- 25 5. An IOH according to any one of claims 1 to 4, in which the inorganic compound is a naturally occurring or a synthetic analogue of a phyllosilicate.
- 30 6. An IOH according to claim 5, in which the naturally occurring or synthetic analogue of a phyllosilicate is a smectite clay.
- 35 7. An IOH according to claim 6, in which the smectite clay is selected from montmorillonite, nontronite, beidellite, volkonskoite, hectorite,

- 62 -

bentonite, saponite, sauconite, magadiite, kenyaite, laponite, vermiculite, synthetic micromica and synthetic hectorite.

5 8. An IOH according to claim 6 or claim 7, in which the naturally occurring phyllosilicate is selected from bentonite, montmorillonite and hectorite.

10 9. An IOH according to any one of claims 5 to 8, in which the phyllosilicate has a platelet thickness less than about 5 nanometers and an aspect ratio greater than about 10:1.

15 10. An IOH according to claim 9, in which the aspect ratio is greater than about 50:1.

11. An IOH according to claim 9 or claim 10, in which the aspect ratio is greater than about 100:1.

20 12. An IOH according to any one of claims 1 to 11, in which the inorganic component includes interlayer or exchangeable metal cations to balance the charge.

25 13. An IOH according to claim 12, in which the metal cation is selected from an alkali metal and alkali earth metal.

30 14. An IOH according to claim 13, in which the alkali or alkali earth metal is selected from Na^+ , K^+ , Mg^{2+} and Ca^{2+} .

35 15. An IOH according to any one of claims 12 to 14, in which the cation exchange capacity of the inorganic component is less than about 400 milli-equivalents per 100 grams.

16. An IOH according to any one of claims 12 to 15, in which the ionic organic component is exchanged with the exchangeable metal ions of the inorganic component.

5 17. An IOH according to any one of claims 1 to 16, in which the ionic species contains onium ion(s).

10 18. An IOH according to claim 17, in which the ionic species containing onium ion(s) is an ammonium, phosphonium or sulfonium derivative of an aliphatic, aromatic or aryl-aliphatic amine, phosphine or sulfide.

15 19. An IOH according to any one of claims 2 to 18, in which the ionic or neutral organic components decompose or sublime endothermically, and/or release volatiles with low combustibility on decomposition and/or induce charring of organic species during thermal decomposition or combustion.

20 20. An IOH according to claim 19, in which the ionic or neutral organic component is a neutral or ionic derivative of a nitrogen based molecule.

25 21. An IOH according to claim 20, in which the nitrogen based molecule is a triazine based species.

22. An IOH according to claim 21, in which the triazine based species is selected from melamine, triphenyl melamine, melam (1,3,5-triazine-2,4,6-triamine-30 n-(4,6-diamino-1,3,5-triazine-yl)), melem ((2,5,8-triamino-1,3,4,6,7,9,9b-heptaazaphenalene)), melon (poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalene-2,5-diyl)imino}), bis and triaziridinyltriazine, trimethylsilyltriazine, melamine cyanurate, melamine phthalate, melamine phosphate, melamine phosphite, melamine phthalimide, dimelamine phosphate, phosphazines, low molecular weight polymers with triazine and

phosphazine repeat units and isocyanuric acid and salts or derivatives thereof.

23. An IOH according to claim 22, in which
5 isocyanuric acid and salts or derivatives thereof are selected from isocyanuric acid, cyanuric acid, triethyl cyanurate, melamine cyanurate, triglycidylcyanurate, triallyl isocyanurate, trichloroisocyanuric acid, 1,3,5-tris(2-hydroxyethyl)triazine-2,4,6-trione,
10 hexamethylenetetramine.melam cyanurate, melem cyanurate and melon cyanurate.

24. An IOH according to any one of claims 19 to 23, in which the organic component is a derivative of
15 phosphoric acid or boric acid.

25. An IOH according to claim 24, in which the derivative of phosphoric acid or boric acid is selected from ammonia polyphosphate, melamine polyphosphate and
20 melamine phosphate ammonium borate.

26. An IOH according to any one of claims 1 to 25, in which the ionic organic component is used in combination with other ionic compounds which are capable
25 of improving compatibility and dispersion between the inorganic and organic components.

27. An IOH according to claim 26, in which the other ionic compound is an amphiphilic molecule that
30 incorporates a hydrophilic ionic group along with hydrophobic alkyl or aromatic moieties.

28. An IOH according to any one of the preceding claims, which further comprises one or more coupling
35 reagents.

- 65 -

29. An IOH according to claim 28, in which the coupling reagent is selected from an organically functionalised silane, zirconate and titanate.

5 30. An IOH according to claim 29, in which the silane coupling reagent is tri-alkoxy, acetoxy or halosilanes functionalised with amino, epoxy, isocyanate, hydroxyl, thiol, mercapto and/or methacryl reactive moieties or modified to incorporate functional groups 10 based on triazine derivatives, long chain alkyl, aromatic or alkylaromatic moieties.

31. A method for the preparation of the IOH defined in any one of claims 1 to 30, which comprises mixing 15 components (i) and (ii) defined in any one of claims 1 to 30 or constituents thereof in one or more steps.

32. A method according to claim 31, in which mixing is achieved using melt, solution or powder processing.

20 33. A method according to claim 31 or claim 32, in which the mixing is achieved using solution processing.

34. Use of the IOH defined in any one of claims 1 to 25 30 as a fire resistant material.

35. A fire resistant formulation which comprises:
(i) the IOH defined in any one of claims 1 to 30; and
30 (ii) one or more flame retardants.

36. A formulation according to claim 35, in which the flame retardant is selected from phosphorus derivatives, nitrogen containing derivatives, molecules 35 containing borate functional groups, molecules containing two or more alcohol groups, molecules which

- 66 -

endothermically release non-combustible decomposition gases and expandable graphite.

37. A formulation according to claim 36, in which 5 the phosphorus derivatives are selected from melamine phosphate, dimelamine phosphate, melamine polyphosphate, ammonia phosphate, ammonia polyphosphate, pentaerythritol phosphate, melamine phosphite and triphenyl phosphine.
- 10 38. A formulation according to claim 36 or claim 37, in which the nitrogen containing derivatives are selected from melamine, melamine cyanurate, melamine phthalate, melamine phthalimide, melam, melem, melon, melam cyanurate, melem cyanurate, melon cyanurate, hexamethylene 15 tetraamine, imidazole, adenine, guanine, cytosine and thymine.
- 20 39. A formulation according to any one of claims 36 to 38, in which the molecules containing borate functional groups are selected from ammonia borate and zinc borate.
- 25 40. A formulation according to any one of claims 36 to 39, in which the molecules containing two or more alcohol groups are selected from pentaerythritol, polyethylene alcohol, polyglycols and carbohydrates.
- 30 41. A formulation according to any one of claims 36 to 40, in which the molecules which endothermically release non-combustible decomposition gases are selected from magnesium hydroxide and aluminum hydroxide.
- 35 42. A method for the preparation of the fire resistant formulation defined in any one of claims 35 to 41, which comprises mixing components (i) and (ii) as defined in any one of claims 1 to 30 or constituents thereof in one or more steps.

- 67 -

43. A method according to claim 42, in which mixing is achieved using melt, solution or powder processing.

44. A method according to claim 42 or claim 43, in 5 which the mixing is achieved using melt processing in a twin screw extruder or batch mixer; or powder processing using a high shear powder mixer or milling procedures.

45. A polyamide fire resistant formulation which 10 comprises either:

(A) (i) the IOH defined in any one of claims 1 to 30; and

(ii) a polyamide based matrix; or

(B) (i) the fire resistant formulation defined 15 in any one of claims 35 to 41; and

(ii) a polyamide based matrix.

46. A formulation according to claim 45, in which 20 the polyamide based matrix comprises generic groups with repeat units based on amides selected from Nylon4, Nylon6, Nylon7, Nylon 11, Nylon12, Nylon46, Nylon66, Nylon 68, Nylon610, Nylon612 and aromatic polyamides and co-polymers, blends or alloys thereof.

25 47. A formulation according to claim 45 or claim 46, in which the polyamide based matrix is selected from Nylon12, Nylon6 and Nylon66 and co-polymers, alloys or blends thereof.

30 48. A formulation according to any one of claims 45 to 47, which further comprises one or more additives.

49. A formulation according to claim 48, in which 35 the additives are selected from polymeric stabilisers; lubricants; antioxidants; pigments, dyes or other additives to alter the materials optical properties or colour; conductive fillers or fibers; release agents; slip

55. A formulation according to any one of claims 48 to 53, in which the polyamide based matrix is present in an amount greater than about 75% w/w, the IOH is present 5 in an amount less than about 3% w/w, the melamine cyanurate flame retardant is present in an amount of about 11 and about 15% w/w, magnesium hydroxide flame retardant present in an amount of about 1 and about 5% w/w and additives are present in an amount less than about 4% w/w.

10

56. A method for the preparation of the polyamide fire resistant formulation defined in any one of claims 45 to 55, which comprises dispersing the IOH as defined in any one of claims 1 to 30 or the fire resistant 15 formulation defined in any one of claims 35 to 41 or constituents thereof into the polyamide based matrix in one or more steps.

20

57. A method according to claim 56, in which at least some of the components are ground prior to mixing.

25

58. A method according to claim 57, in which the components are ground to a particle size less than about 200 microns.

59. A method according to claim 57 or claim 58, in which dispersion is achieved using melt, solution or powder processing.

30

60. A method according to any one of claims 57 to 59, in which the dispersion is achieved using melt processing in a single or twin screw extruder, batch mixer or continuous compounder.

35

61. A method according to claim 60, in which the melt processing is conducted in a twin screw extruder.

- 70 -

62. A method according to any one of claims 56 to 51, in which the dispersion occurs at a sufficient shear rate, shear stress and residence time to disperse the IOH at least partially on a nanometer scale.

5

63. A fire resistant article or parts thereof which is composed wholly or partly of the IOH as defined in any one of claims 1 to 30 and/or fire resistant formulation defined in any one of claims 35 to 41 and claims 45 to 55.

10

64. A fire resistant article or parts thereof as defined in claim 63, which is used in transport, building, construction, electrical or optical applications.

15 65. A fire resistant article or parts thereof as defined in claim 64, in which the transport application is air, automotive, aerospace or nautical.

20 66. A fire resistant article or parts thereof as defined in any one of claims 63 to 65, which is a hollow article or sheet.

25 67. A fire resistant article or parts thereof as defined in any one of claims 63 to 66 which is selected from pipes, ducts, fabric, carpet, cables, wires, fibres, Environmental control systems, stowage bin hinge covers, cable trays, ECS duct spuds, latches, brackets, passenger surface units and thermoplastic laminate sheet.

30 68. A fire resistant hollow article or parts thereof which is composed wholly or partly of the fire resistant formulation defined in claim 54 or claim 55 and manufactured by rotational moulding or extrusion.

35 69. A fire resistant fibre, fabric, carpet or parts thereof which is composed wholly or partly of the fire

- 71 -

resistant formulation defined in claim 54 or claim 55 and manufactured by melt spinning or extrusion.

70. A fire resistant article or parts thereof which
5 is composed wholly or partly of the formulation defined in
claim 54 or claim 55 and manufactured by sintering.

71. A fire resistant article or parts thereof which
is composed wholly or partly of the fire resistant
10 formulation defined in claim 54 or claim 55 and
manufactured by injection or compression moulding.

72. A method of preparing the fire resistant article
or parts thereof defined in any one of claims 59 to 71,
15 which comprises moulding or forming the IOH as defined in
any one of claims 1 to 30 and/or the fire resistant
formulation or constituents thereof as defined in any one
of claims 35 to 41 and claims 45 to 51.

20 73. A method according to claim 72, in which the
moulding or forming is carried out using extrusion,
injection moulding, compression moulding, rotational
moulding, blow moulding, sintering, thermoforming,
calendering or combinations thereof.

REC'D 28 FEB 2005

WIPO

PCT

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference FP18623	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416).
International Application No. PCT/AU2003/001443	International Filing Date (day/month/year) 31 October 2003	Priority Date (day/month/year) 31 October 2002
International Patent Classification (IPC) or national classification and IPC Int. Cl. ⁷ C09K 21/06, C01B 33/44, C08K 3/34		
Applicant COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 3 sheets, including this cover sheet.

This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 13 sheet(s).

3. This report contains indications relating to the following items:

- I Basis of the report
- II Priority
- III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV Lack of unity of invention
- V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI Certain documents cited
- VII Certain defects in the international application
- VIII Certain observations on the international application

Date of submission of the demand 28 May 2004	Date of completion of the report 22 February 2005
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer GAYE HOROBIN Telephone No. (02) 6283 2069

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/AU2003/001443

I. Basis of the report

1. With regard to the elements of the international application:*

the international application as originally filed.

the description, pages 1-5,7,8,10-60, as originally filed,
pages , filed with the demand,
pages 6,9, received on 25 January 2005 with the letter of 25 January 2005

the claims, pages , as originally filed,
pages , as amended (together with any statement) under Article 19,
pages , filed with the demand,
pages 61-71, received on 25 January 2005 with the letter of 25 January 2005

the drawings, pages 1/6-6/6, as originally filed,
pages , filed with the demand,
pages , received on with the letter of

the sequence listing part of the description:
pages , as originally filed
pages , filed with the demand
pages , received on with the letter of

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language which is:

the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

the language of publication of the international application (under Rule 48.3(b)).

the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

contained in the international application in written form.

filed together with the international application in computer readable form.

furnished subsequently to this Authority in written form.

furnished subsequently to this Authority in computer readable form.

The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

4. The amendments have resulted in the cancellation of:

the description, pages

the claims, Nos.

the drawings, sheets/fig.

5. This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

** Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/AU2003/001443

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims 1-71	YES
	Claims	NO
Inventive step (IS)	Claims 1-71	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-71	YES
	Claims	NO

2. Citations and explanations (Rule 70.7)

NOVELTY(N), INVENTIVE STEP(IS)

No citation or obvious combination of citations discloses the combination of at least one ionic organic compound and one or more neutral organic compounds intercalated between the layers of an expandable or swellable layered inorganic component.

- 6 -

Accordingly, there is a need for the development of new flame retarding systems which both meet the performance criteria associated with specific applications and address the above concerns.

5

SUMMARY OF THE INVENTION

According to one aspect of the present invention there is provided an inorganic-organic hybrid (IOH) which comprises:

10

(i) an expandable or swellable layered inorganic component; and

(ii) an organic component including at least one ionic organic component and one or more neutral organic components which are intercalated between and/or

15

associated with the layer(s) of the inorganic component,

the ionic or neutral organic components being capable of decomposing or subliming endothermically, and/or releasing volatiles with low combustibility on decomposition and/or inducing charring of organic species

20

during thermal decomposition or combustion.

According to another aspect of the present invention there is provided a method for the preparation of the IOH defined above which comprises mixing components (i) and (ii) defined above or constituents thereof in one or more steps.

The present invention also provides the use of the IOH defined above as a fire resistant material.

According to a further aspect of the present invention there is provided a fire resistant formulation which comprises:

25

(i) the IOH defined above; and

(ii) one or more flame retardants.

According to a still further aspect of the present invention there is provided a method for the preparation of the fire resistant formulation defined above which comprises mixing components (i) and (ii) as defined above or constituents thereof in one or more steps.

30

The present invention also provides a polyamide fire resistant formulation which comprises either:

- 9 -

of the inorganic component and/or one or more coupling reagents.

The term "associated with" is used herein in its broadest sense and refers to the neutral organic component 5 being attached to the layer(s) of the inorganic component, for example, by secondary bonding interactions, such as, Van der Waals interactions or hydrogen bonding or trapped by steric limitation.

Suitable examples of ionic species include those 10 that contain onium ions such as ammonium (primary, secondary, tertiary and quaternary), phosphonium or sulfonium derivatives of aliphatic, aromatic or aryl-aliphatic amines, phosphines and sulfides.

Such compounds may be prepared by any method 15 known to those skilled in the art. For example, salts prepared by acid-base type reactions with mineral or organic acids including hydrochloric, sulfuric, nitric, phosphoric, acetic and formic acids, by Lewis-acid - Lewis-base type reactions or by reaction with alkyl 20 halides to form quaternary salts for example using Menschutkin type methodology.

Ionic or neutral compounds which are known to decompose or sublime endothermically, and/or which release volatiles with low combustibility on decomposition and/or 25 induce charring of organic species during thermal decomposition or combustion are used in the IOH of the present invention.

Suitable species include neutral or ionic 30 derivatives of nitrogen based molecules, such as, triazine based species, for example, melamine, triphenyl melamine, melam (1,3,5-triazine-2,4,6-triamine-n-(4,6-diamino-1,3,5-triazine-yl)), melem ((-2,5,8-triamino-1,3,4,6,7,9,9b-heptaazaphenalenene)), melon (poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalenene-2,5-diyl)imino}), bis and 35 triaziridinyltriazine, trimethylsilyltriazine, melamine cyanurate, melamine phthalate, melamine phosphate, melamine phosphite, melamine phthalimide, dimelamine phosphate, phosphazines and/or low molecular weight

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An inorganic-organic hybrid (IOH) which comprises:

5 (i) an expandable or swellable layered inorganic component; and
(ii) an organic component including at least one ionic organic component and one or more neutral organic components which are intercalated between and/or 10 associated with the layer(s) of the inorganic component, the ionic or neutral organic components being capable of decomposing or subliming endothermically, and/or releasing volatiles with low combustibility on decomposition and/or inducing charring of organic species 15 during thermal decomposition or combustion.

2. An IOH according to claim 1, in which the inorganic component is rendered positively or negatively charged due to isomorphic substitution of elements within 20 the layers.

3. An IOH according to claim 1 or claim 2, in which the inorganic component is selected from a 1:1 layered silicate structure, a 2:1 layered silicate structure, a 25 double hydroxide of the general formula $Mg_6Al_{3.4}(OH)_{18.8}(CO_3)_{1.7}\cdot H_2O$ and a synthetically prepared layered material.

4. An IOH according to any one of claims 1 to 3, in 30 which the inorganic compound is a naturally occurring or a synthetic analogue of a phyllosilicate.

5. An IOH according to claim 4, in which the naturally occurring or synthetic analogue of a 35 phyllosilicate is a smectite clay.

6. An IOH according to claim 5, in which the smectite clay is selected from montmorillonite, nontronite, beidellite, volkonskoite, hectorite, bentonite, saponite, saucnite, magadiite, kenyait, 5 laponite, vermiculite, synthetic micromica and synthetic hectorite.

7. An IOH according to claim 5 or claim 8, in which the naturally occurring phyllosilicate is selected from 10 bentonite, montmorillonite and hectorite.

8. An IOH according to any one of claims 4 to 7, in which the phyllosilicate has a platelet thickness less than about 5 nanometers and an aspect ratio greater than 15 about 10:1.

9. An IOH according to claim 8, in which the aspect ratio is greater than about 50:1.

20 10. An IOH according to claim 8 or claim 9, in which the aspect ratio is greater than about 100:1.

11. An IOH according to any one of claims 1 to 10, in which the inorganic component includes interlayer or 25 exchangeable metal cations to balance the charge.

12. An IOH according to claim 11, in which the metal cation is selected from an alkali metal and alkali earth metal.

30 13. An IOH according to claim 12, in which the alkali or alkali earth metal is selected from Na^+ , K^+ , Mg^{2+} and Ca^{2+} .

35 14. An IOH according to any one of claims 11 to 13, in which the cation exchange capacity of the inorganic

component is less than about 400 milli-equivalents per 100 grams.

15. An IOH according to any one of claims 11 to 14,
5 in which the ionic organic component is exchanged with the
exchangeable metal ions of the inorganic component.

16. An IOH according to any one of claims 1 to 15,
10 in which the ionic species contains onium ion(s).

17. An IOH according to claim 16, in which the ionic
species containing onium ion(s) is an ammonium,
15 phosphonium or sulfonium derivative of an aliphatic,
aromatic or aryl-aliphatic amine, phosphine or sulfide.

18. An IOH according to any one of claims 1 to 17,
20 in which the ionic or neutral organic component is a
neutral or ionic derivative of a nitrogen based molecule.

19. An IOH according to claim 18, in which the
nitrogen based molecule is a triazine based species.

20. An IOH according to claim 19, in which the
25 triazine based species is selected from melamine,
triphenyl melamine, melam (1,3,5-triazine-2,4,6-triamine-
n-(4,6-diamino-1,3,5-triazine-yl)), melem ((-2,5,8-
triarnino-1,3,4,6,7,9,9b-heptaazaphenalenene)), melon
(poly{8-amino-1,3,4,6,7,9,9b-heptaazaphenalenene-2,5-
30 diyl)imino}), bis and triaziridinyltriazine,
trimethylsilyltriazine, melamine cyanurate, melamine
phthalate, melamine phosphate, melamine phosphite,
melamine phthalimide, dimelamine phosphate, phosphazines,
low molecular weight polymers with triazine and
phosphazine repeat units and isocyanuric acid and salts or
35 derivatives thereof.

- 64 -

21. An IOH according to claim 20, in which isocyanuric acid and salts or derivatives thereof are selected from isocyanuric acid, cyanuric acid, triethyl cyanurate, melamine cyanurate, triglycidylcyanurate, 5 triallyl isocyanurate, trichloroisocyanuric acid, 1,3,5-tris(2-hydroxyethyl)triazine-2,4,6-trione, hexamethylenetetramine.melam cyanurate, melem cyanurate and melon cyanurate.

10 22. An IOH according to any one of claims 18 to 21, in which the organic component is a derivative of phosphoric acid or boric acid.

15 23. An IOH according to claim 22, in which the derivative of phosphoric acid or boric acid is selected from ammonia polyphosphate, melamine polyphosphate and melamine phosphate ammonium borate.

20 24. An IOH according to any one of claims 1 to 23, in which the ionic organic component is used in combination with other ionic compounds which are capable of improving compatibility and dispersion between the inorganic and organic components.

25 25. An IOH according to claim 24, in which the other ionic compound is an amphiphilic molecule that incorporates a hydrophilic ionic group along with hydrophobic alkyl or aromatic moieties.

30 26. An IOH according to any one of the preceding claims, which further comprises one or more coupling reagents.

35 27. An IOH according to claim 26, in which the coupling reagent is selected from an organically functionalised silane, zirconate and titanate.

- 65 -

28. An IOH according to claim 27, in which the silane coupling reagent is tri-alkoxy, acetoxy or halosilanes functionalised with amino, epoxy, isocyanate, hydroxyl, thiol, mercapto and/or methacryl reactive moieties or modified to incorporate functional groups based on triazine derivatives, long chain alkyl, aromatic or alkylaromatic moieties.

5

29. A method for the preparation of the IOH defined in any one of claims 1 to 28, which comprises mixing components (i) and (ii) defined in any one of claims 1 to 28 or constituents thereof in one or more steps.

10

30. A method according to claim 29, in which mixing is achieved using melt, solution or powder processing.

15

31. A method according to claim 29 or claim 30, in which the mixing is achieved using solution processing.

20

32. Use of the IOH defined in any one of claims 1 to 28 as a fire resistant material.

25

33. A fire resistant formulation which comprises:

(i) the IOH defined in any one of claims 1 to 28; and

(ii) one or more flame retardants.

30

34. A formulation according to claim 33, in which the flame retardant is selected from phosphorus derivatives, nitrogen containing derivatives, molecules containing borate functional groups, molecules containing two or more alcohol groups, molecules which endothermically release non-combustible decomposition gases and expandable graphite.

35

35. A formulation according to claim 34, in which the phosphorus derivatives are selected from melamine

- 66 -

phosphate, dimelamine phosphate, melamine polyphosphate, ammonia phosphate, ammonia polyphosphate, pentaerythritol phosphate, melamine phosphite and triphenyl phosphine.

5 36. A formulation according to claim 34 or claim 35, in which the nitrogen containing derivatives are selected from melamine, melamine cyanurate, melamine phthalate, melamine phthalimide, melam, melem, melon, melam cyanurate, melem cyanurate, melon cyanurate, hexamethylene tetraamine, imidazole, adenine, guanine, cytosine and thymine.

10 37. A formulation according to any one of claims 34 to 36, in which the molecules containing borate functional groups are selected from ammonia borate and zinc borate.

15 38. A formulation according to any one of claims 34 to 37, in which the molecules containing two or more alcohol groups are selected from pentaerythritol, polyethylene alcohol, polyglycols and carbohydrates.

20 39. A formulation according to any one of claims 34 to 38, in which the molecules which endothermically release non-combustible decomposition gases are selected from magnesium hydroxide and aluminum hydroxide.

25 40. A method for the preparation of the fire resistant formulation defined in any one of claims 33 to 39, which comprises mixing components (i) and (ii) as defined in any one of claims 1 to 28 or constituents thereof in one or more steps.

30 41. A method according to claim 40, in which mixing is achieved using melt, solution or powder processing.

35 42. A method according to claim 40 or claim 41, in which the mixing is achieved using melt processing in a

twin screw extruder or batch mixer; or powder processing using a high shear powder mixer or milling procedures.

43. A polyamide fire resistant formulation which

5 comprises either:

(A) (i) the IOP defined in any one of claims 1 to 28; and

(ii) a polyamide based matrix; or

(B) (i) the fire resistant formulation defined 10 in any one of claims 33 to 39; and

(ii) a polyamide based matrix.

44. A formulation according to claim 43, in which

the polyamide based matrix comprises generic groups with 15 repeat units based on amides selected from Nylon4, Nylon6, Nylon7, Nylon 11, Nylon12, Nylon46, Nylon66, Nylon 68, Nylon610, Nylon612 and aromatic polyamides and co-polymers, blends or alloys thereof.

20 45. A formulation according to claim 43 or claim 44, in which the polyamide based matrix is selected from Nylon12, Nylon6 and Nylon66 and co-polymers, alloys or blends thereof.

25 46. A formulation according to any one of claims 43 to 45, which further comprises one or more additives.

47. A formulation according to claim 46, in which 30 the additives are selected from polymeric stabilisers; lubricants; antioxidants; pigments, dyes or other additives to alter the materials optical properties or colour; conductive fillers or fibers; release agents; slip agents; plasticisers; antibacterial or fungal agents; and processing agents.

- 68 -

48. A formulation according to claim 47, in which the polymeric stabiliser is a UV, light or thermal stabilizer.

5 49. A formulation according to claim 47 or claim 48, in which the processing agents are selected from dispersing reagents, foaming or blowing agents, surfactants, waxes, coupling reagents, rheology modifiers, film forming reagents and free radical generating
10 reagents.

50. A formulation according to any one of claims 43 to 49, in which the polyamide based matrix is Nylon12, Nylon6 and/or Nylon66; the IOH is montmorillonite or
15 hectorite modified with melamine hydrochloride and/or melamine cyanurate hydrochloride and/or melamine and/or melamine cyanurate; and the flame retardant is melamine cyanurate and/or magnesium hydroxide; and the additive is a processing agent and/or a polymeric stabiliser.

20 51. A formulation according to any one of claims 46 to 50, in which the polyamide based matrix is present in an amount of about 45 to about 95% w/w, the IOH is present in an amount less than about 25% w/w and the flame
25 retardant and/or additives are present in an amount less than about 30% w/w.

52. A formulation according to any one of claims 46 to 51, in which the polyamide based matrix is present in
30 an amount greater than about 75% w/w, the IOH is present in an amount less than about 3% w/w, the melamine cyanurate flame retardant is present in an amount of about 11 to about 15% w/w and additives are present in an amount of about less than about 4% w/w.

35 53. A formulation according to any one of claims 46 to 51, in which the polyamide based matrix is present in

an amount greater than about 75% w/w, the IOH is present in an amount less than about 3% w/w, the melamine cyanurate flame retardant is present in an amount of about 11 and about 15% w/w, magnesium hydroxide flame retardant 5 present in an amount of about 1 and about 5% w/w and additives are present in an amount less than about 4% w/w.

54. A method for the preparation of the polyamide fire resistant formulation defined in any one of claims 43 10 to 53, which comprises dispersing the IOH as defined in any one of claims 1 to 28 or the fire resistant formulation defined in any one of claims 33 to 39 or constituents thereof into the polyamide based matrix in one or more steps.

15

55. A method according to claim 54, in which at least some of the components are ground prior to mixing.

56. A method according to claim 55, in which the 20 components are ground to a particle size less than about 200 microns.

57. A method according to claim 55 or claim 56, in which dispersion is achieved using melt, solution or 25 powder processing.

58. A method according to any one of claims 55 to 57, in which the dispersion is achieved using melt processing in a single or twin screw extruder, batch mixer 30 or continuous compounder.

59. A method according to claim 58, in which the melt processing is conducted in a twin screw extruder.

35 60. A method according to any one of claims 54 to 59, in which the dispersion occurs at a sufficient shear

- 70 -

rate, shear stress and residence time to disperse the IOH at least partially on a nanometer scale.

61. A fire resistant article or parts thereof which
5 is composed wholly or partly of the IOH as defined in any one of claims 1 to 28 and/or fire resistant formulation defined in any one of claims 33 to 39 and claims 43 to 53.

62. A fire resistant article or parts thereof as
10 defined in claim 61, which is used in transport, building, construction, electrical or optical applications.

63. A fire resistant article or parts thereof as
defined in claim 62, in which the transport application is
15 air, automotive, aerospace or nautical.

64. A fire resistant article or parts thereof as
defined in any one of claims 61 to 63, which is a hollow article or sheet.

20
65. A fire resistant article or parts thereof as
defined in any one of claims 61 to 64 which is selected from pipes, ducts, fabric, carpet, cables, wires, fibres, Environmental control systems, stowage bin hinge covers, 25 cable trays, ECS duct spuds, latches, brackets, passenger surface units and thermoplastic laminate sheet.

66. A fire resistant hollow article or parts thereof which is composed wholly or partly of the fire resistant 30 formulation defined in claim 52 or claim 53 and manufactured by rotational moulding or extrusion.

67. A fire resistant fibre, fabric, carpet or parts thereof which is composed wholly or partly of the fire 35 resistant formulation defined in claim 52 or claim 53 and manufactured by melt spinning or extrusion.

68. A fire resistant article or parts thereof which is composed wholly or partly of the formulation defined in claim 52 or claim 53 and manufactured by sintering.

5 69. A fire resistant article or parts thereof which is composed wholly or partly of the fire resistant formulation defined in claim 52 or claim 53 and manufactured by injection or compression moulding.

10 70. A method of preparing the fire resistant article or parts thereof defined in any one of claims 57 to 69, which comprises moulding or forming the IOH as defined in any one of claims 1 to 28 and/or the fire resistant formulation or constituents thereof as defined in any one 15 of claims 33 to 39 and claims 43 to 49.

71. A method according to claim 70, in which the moulding or forming is carried out using extrusion, injection moulding, compression moulding, rotational 20 moulding, blow moulding, sintering, thermoforming, calendering or combinations thereof.

PATENT COOPERATION TREATY

WO 2004/039916 2005
PCT/AU2003/00142

EV336518308US

PCT

NOTICE INFORMING THE APPLICANT OF THE
COMMUNICATION OF THE INTERNATIONAL
APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

Date of mailing (day/month/year)
13 May 2004 (13.05.2004)Applicant's or agent's file reference
FP18623International application No.
PCT/AU2003/001443International filing date (day/month/year)
31 October 2003 (31.10.2003)

GRIFFITH HACK

31 MAY 2004

1 ... REC'd.
2 ... DAB
3 ...

IMPORTANT NOTICE

Priority date (day/month/year)
31 October 2002 (31.10.2002)

Applicant

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this notice:

AU, AZ, BY, CH, CN, CO, DZ, EP, HU, JP, KG, KP, KR, MD, MK, MZ, RU, TM, US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE, AG, AL, AM, AP, AT, BA, BB, BG, BR, BW, BZ, CA, CR, CU, CZ, DE, DK, DM, EA, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, ID, IL, IN, IS, KE, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MG, MN, MW, MX, NI, NO, NZ, OA, OM, PG, PH, PL, PT, RO, SC, SD, SE, SG, SK, SL, SY, TJ, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this notice is a copy of the international application as published by the International Bureau on
13 May 2004 (13.05.2004) under No. WO 2004/039916

4. TIME LIMITS for filing a demand for international preliminary examination and for entry into the national phase

The applicable time limit for entering the national phase will, subject to what is said in the following paragraph, be 30 MONTHS from the priority date, not only in respect of any elected Office if a demand for international preliminary examination is filed before the expiration of 19 months from the priority date, but also in respect of any designated Office, in the absence of filing of such demand, where Article 22(1) as modified with effect from 1 April 2002 applies in respect of that designated Office. For further details, see *PCT Gazette* No. 44/2001 of 1 November 2001, pages 19926, 19932 and 19934, as well as the *PCT Newsletter*, October and November 2001 and February 2002 issues.

In practice, time limits other than the 30-month time limit will continue to apply, for various periods of time, in respect of certain designated or elected Offices. For regular updates on the applicable time limits (20, 21, 30 or 31 months, or other time limit), Office by Office, refer to the *PCT Gazette*, the *PCT Newsletter* and the *PCT Applicant's Guide*, Volume II, National Chapters, all available from WIPO's Internet site, at <http://www.wipo.int/pct/en/index.html>.

For filing a demand for international preliminary examination, see the *PCT Applicant's Guide*, Volume I/A, Chapter IX. Only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination (at present, all PCT Contracting States are bound by Chapter II).

It is the applicant's sole responsibility to monitor all these time limits.

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Authorized officer

Dorothée Mühlhausen

Facsimile No. +41 22 740 14 35

PCT/IB/308 (April 2002)

Facsimile No. +41 22 338 87 40

PATENT COOPERATION TREATY

Recording

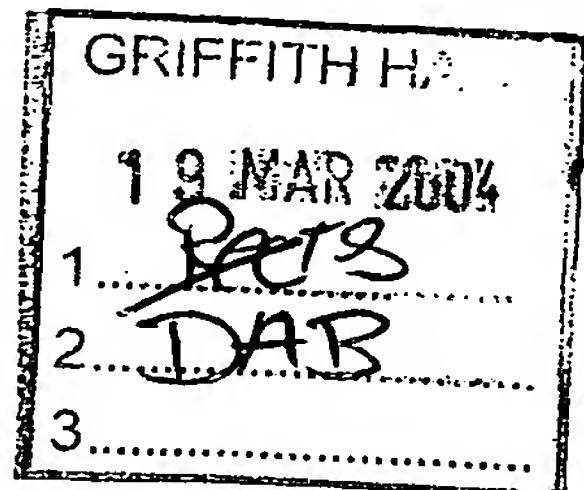
02 MAY 2005

PCT

NOTIFICATION OF THE RECORDING
OF A CHANGE(PCT Rule 92bis.1 and
Administrative Instructions, Section 422)Date of mailing (day/month/year)
02 March 2004 (02.03.2004)

From the INTERNATIONAL BUREAU

To:

GRIFFITH HACK
509 St Kilda Road
Melbourne, Victoria 3004
AustraliaApplicant's or agent's file reference
FP18623

IMPORTANT NOTIFICATION

International application No.
PCT/AU2003/001443

FIRE RESISTANT

International filing date (day/month/year)
31 October 2003 (31.10.2003)

1. The following indications appeared on record concerning:

 the applicant the inventor the agent the common representative

Name and Address

BATEMAN, Stuart, Arthur
3 Wortley Avenue
Mount Waverley, Victoria 3149
Australia

State of Nationality

AU

State of Residence

AU

Telephone No.

Facsimile No.

Teleprinter No.

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

 the person the name the address the nationality the residence

Name and Address

State of Nationality

State of Residence

Telephone No.

Facsimile No.

Teleprinter No.

3. Further observations, if necessary:

The person indicated in Box 1 has changed from applicant/inventor for all Designated States to applicant/inventor for the United States of America only.

4. A copy of this notification has been sent to:

<input checked="" type="checkbox"/> the receiving Office	<input type="checkbox"/> the designated Offices concerned
<input type="checkbox"/> the International Searching Authority	<input type="checkbox"/> the elected Offices concerned
<input type="checkbox"/> the International Preliminary Examining Authority	<input type="checkbox"/> other:

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Authorized officer

Ana MENA VALENCIA

Facsimile No. (41-22) 338.87.40

Telephone No. (41-22) 338 8665